

IN THE UNITED STATES DISTRICT COURT  
FOR THE DISTRICT OF DELAWARE

THE GILLETTE COMPANY,	)	
	)	
Plaintiff,	)	
	)	
v.	)	C.A. No. 15-1158 (LPS)
	)	
DOLLAR SHAVE CLUB, INC., DORCO	)	
COMPANY LTD. and PACE SHAVE, INC.	)	
	)	
Defendants.	)	

**JOINT CLAIM CONSTRUCTION STATEMENT**

Pursuant to the Court's Scheduling Order [D.I. 204], the parties hereby submit the Joint Claim Construction Chart attached as Exhibit A, setting forth (i) the claim terms and phrases in dispute; (ii) the parties' proposed constructions; and (iii) the intrinsic evidence the parties rely on to support their proposed constructions. Attached as Exhibit B is a copy of The Gillette Company's asserted patent, U.S. Patent No. 6,684,513 ("the '513 patent"). Attached as Exhibit C are excerpts from the '513 patent file history identified by the parties as intrinsic evidence supporting their proposed constructions. Exhibit C also includes a copy of U.S. Patent No. 5,795,648, which Gillette contends is part of the certified file history and Defendants Pace Shave and Dollar Shave Club have not had an opportunity to verify. Attached are also Exhibit D, a copy of U.S. Patent No. 3,911,579, also part of the file history; and Exhibit E, excerpts from Mattox, "Handbook of Physical Vapor Deposition (PVD) Processing" (1998), a handbook cited in the '513 patent. Each party may cite additional or different intrinsic evidence, and may rely on the intrinsic evidence cited by the other party. Either party may also rely on extrinsic evidence as may be appropriate.

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# EXHIBIT A

***The Gillette Company v. Dollar Shave Club, Inc. et al.***  
**District of Delaware, C.A. No. 15-1158-LPS-CJB**  
**Joint Claim Construction Chart**

<b>'513 Patent Term</b>	<b>AGREED CONSTRUCTION</b>
diamond-like carbon material (claims 2, 31)	An amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond

**DISPUTED CONSTRUCTIONS**

<b>'513 Patent Term</b>	<b>PLAINTIFF'S PROPOSED CONSTRUCTION</b>	<b>DEFENDANTS PACE SHAVE/DSC'S PROPOSED CONSTRUCTION</b>
amorphous material (claims 1, 6, 20, 24)	<p>material lacking long-range crystalline order</p> <p><b>'513 Patent:</b> Claims 1-2, 6, 8, 15-17, 19-20, 23-24; Col. 1:9-14; Col. 1:45-49; Col. 2:51-67; Col. 3:1-4.</p> <p><b>'513 Patent File History:</b> Resp. to O.A. dated 10/19/2001 at 4; Resp. to O.A. dated 6/6/2002 at 3; Resp. to O.A. dated 1/8/2003 at 3; Resp. to O.A. dated 8/6/2003 at 8; U.S. Patent No. 5,795,648, Col. 4:38-43<sup>1</sup>.)</p> <p>Mattox, "Handbook of Physical Vapor Deposition (PVD) Processing" (1998) (cited at '513 patent, col. 2:57-3:4), at 487-488, 511-513, 767, 807.</p>	<p>material having no detectable crystal structure</p> <p><b>'513 Patent:</b> Fig. 1, Col. 1:9-14, 1:46-51, 2:29-30, 2:37-39, 2:52-3:4, Claims 1, 6, 20, 24</p> <p>Mattox, "Handbook of Physical Vapor Deposition (PVD) Processing" (1998) (cited at '513 patent, col. 2:57-3:4): pages 487-488, 511-513, 767, 807</p> <p><b>'513 File History:</b>  10/19/2001 Response at 4-5;  2/4/2002 O.A. at 3-4;  6/6/2002 Response at 3-5;  8/8/2002 O.A. at 2-6;  1/8/2003 Response at 3-5;  8/6/2003 Response at 8-11</p>

<sup>1</sup> Plaintiff The Gillette Company contends that the '648 patent is intrinsic evidence because it is included as part of the certified file history (Ex. C at GILLETTE-DSC-0220865). Defendants Pace Shave and Dollar Shave Club reserve their rights with regard to this reference having just received the Plaintiff's citation.



'513 Patent Term	PLAINTIFF'S PROPOSED CONSTRUCTION	DEFENDANTS PACE SHAVE/DSC'S PROPOSED CONSTRUCTION
<p>carbon containing material, doped with another element (claim 28, 35)</p> <p>wherein said hard coating is doped with another element (claims 19, 23)</p>	<p>carbon containing material, with another element added</p> <p>wherein the hard coating has another element added</p> <p><b>'513 Patent:</b> Claims 1, 19, 28-31, 35-37; Col. 2:57-62.</p> <p><b>'513 Patent File History:</b> U.S. Patent No. 5,795,648, Col. 4:50-62, Col. 5:9-33, Col. 6:56-Col. 7:6.)</p>	<p>carbon containing material, whereby another element is introduced into the material in small amounts to modify certain properties of the material</p> <p>wherein another element is introduced into the hard coating in small amounts to modify certain properties of the coating</p> <p><b>'513 Patent:</b> Col. 2:52-63, Claims 19, 23, 28, 35</p> <p><b>'513 File History:</b> Application page 3; 8/6/2003 Response at 8, 11</p>
<p>overcoat layer of a chromium containing material (claims 1, 20, 24, 28, 35)</p>	<p>No construction is needed.</p> <p>To the extent the Court believes a construction is necessary, Plaintiff will propose the ordinary meaning of the term as "a layer of chromium containing material on top of the layer of [hard coating]" (for claims 1, 20, 24) / [hard carbon containing material] (for claims 28, 35)."</p> <p><b>'513 Patent:</b> Abstract; Claims 1-38; Figure 1; Col. 1:32-68; Col. 2:1-7, 13-22, 36-39; Col. 3:5-14, 31-48, 63-67.</p>	<p>intermediate layer of chromium containing material that improves adhesion to the layer of [hard coating (for claims 1, 20, 24) /hard carbon containing material (for claims 28, 35)]</p> <p><b>'513 Patent:</b> Abstract, Fig. 1, Col. 1:16-19, 1:31-44, 1:51-57, 1:64-66, 2:14-16, 2:29-30, 2:37-39, 2:46-49, 3:5-14, 3:36-45, 3:64-67, Claims 1, 20, 24, 28, 35</p> <p><b>'513 File History:</b> 5/22/2001 O.A. at 3-9; 10/19/2001 Response at 5; 2/4/2002 O.A. at 3-4; 6/6/2002 Response at 3-4; 8/8/2002 O.A. at 3; 1/8/2003 Response at 4; 8/6/2003 Response at 9-10</p> <p><b>Lane (US Patent No. 3,911,579)</b> (cited and discussed in several rejections and</p>

<b>'513 Patent Term</b>	<b>PLAINTIFF'S PROPOSED CONSTRUCTION</b>	<b>DEFENDANTS PACE SHAVE/DSC'S PROPOSED CONSTRUCTION</b>
		responses in '513 file history): Abstract, Figs. 1, 5, Col. 3:23-29, 3:36- 40, 4:3-5, 4:13-15, 6:11-19, 18:18-25, Claims 1, 18, 33

# EXHIBIT B







US006684513B1

(12) **United States Patent**  
**Clipstone et al.**

(10) **Patent No.:** **US 6,684,513 B1**  
 (45) **Date of Patent:** **Feb. 3, 2004**

(54) **RAZOR BLADE TECHNOLOGY**

(75) Inventors: **Colln John Clipstone**, Weston, MA (US); **Steve Hahn**, Wellesley, MA (US); **Neville Sonnenberg**, Newton, MA (US); **Charles White**, Lynnfield, MA (US); **Andrew Zhuk**, Acton, MA (US)

(73) Assignee: **The Gillette Company**, Boston, MA (US)

(\*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **09/515,421**

(22) Filed: **Feb. 29, 2000**

(51) Int. Cl.<sup>7</sup> ..... **B26B 21/58**; **B26B 21/60**

(52) U.S. Cl. .... **30/346.54**; **30/346.53**

(58) Field of Search ..... **30/50**, **346.53**, **30/346.54**, **346.55**, **346**, **76/DIG. 8**

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International Search Report PCT/US01/06206.

\* cited by examiner

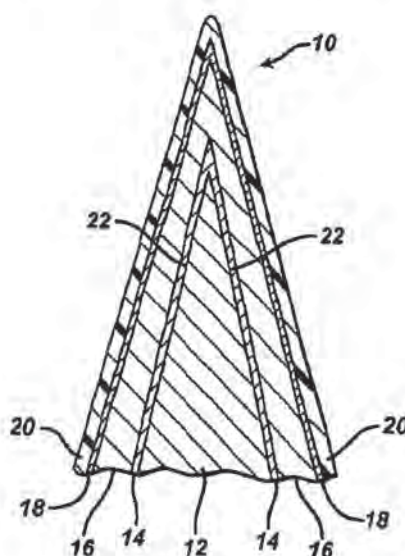
*Primary Examiner*—Hwei-Siu Payer

(74) *Attorney, Agent, or Firm*—Fish & Richardson P.C.

(57) **ABSTRACT**

A razor blade including a substrate with a cutting edge defined by a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of a chromium containing material on the layer of hard carbon coating, and an outer layer of polytetrafluoroethylene coating over the overcoat layer.

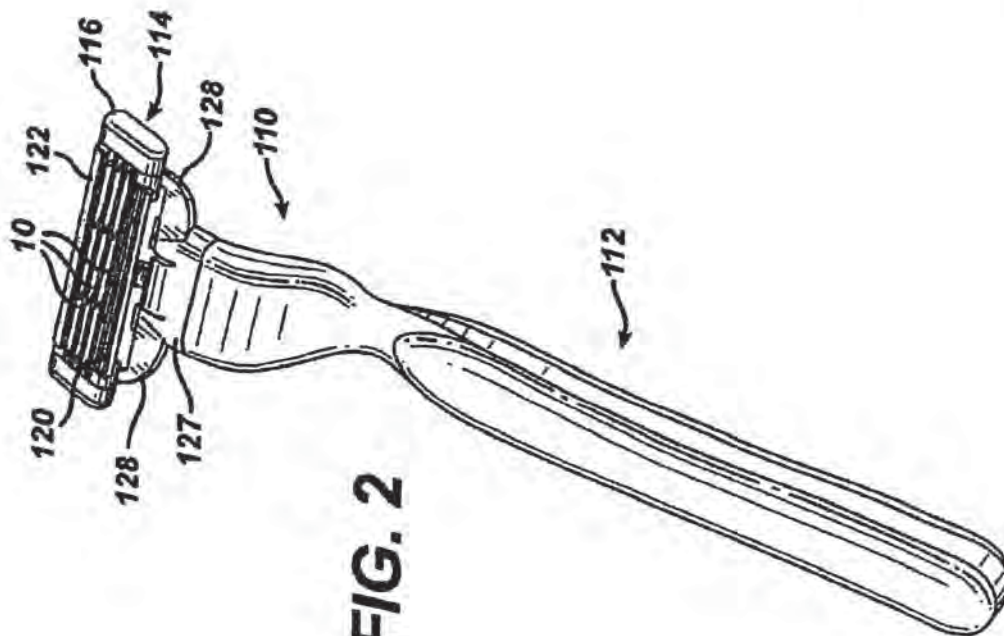
**38 Claims, 1 Drawing Sheet**



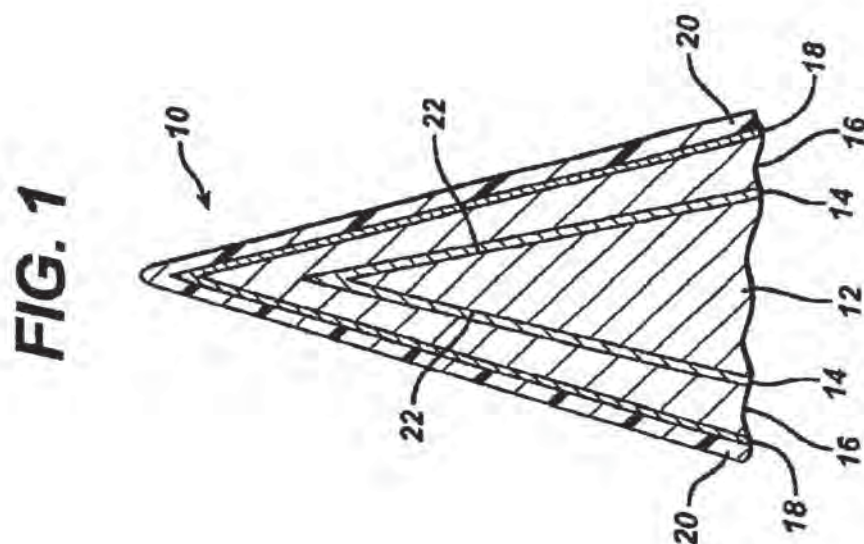
## U.S. Patent

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**FIG. 2**



**FIG. 1**



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## RAZOR BLADE TECHNOLOGY

The invention relates to improvements to razors and razor blades.

A razor blade is typically formed of a suitable substrate material such as stainless steel, and a cutting edge is formed with a wedge-shaped configuration with an ultimate tip having a radius less than about 1000 angstroms, e.g., about 200–300 angstroms. Hard coatings such as diamond, amorphous diamond, diamond-like carbon (DLC) material, nitrides, carbides, oxides or ceramics are often used to improve strength, corrosion resistance and shaving ability, maintaining needed strength while permitting thinner edges with lower cutting forces to be used. Polytetrafluoroethylene (PTFE) outer layer can be used to provide friction reduction. Interlayers of niobium or chromium containing materials can aid in improving the binding between the substrate, typically stainless steel, and hard carbon coatings, such as DLC. Examples of razor blade cutting edge structures and processes of manufacture are described in U.S. Pat. Nos. 5,295,305; 5,232,568; 4,933,058; 5,032,243; 5,497,550; 5,940,975; 5,669,144; EP 0591339; and PCT 92/03330, which are hereby incorporated by reference.

In use, the ultimate tip of the edges having hard coatings and polytetrafluoroethylene outer layers can become more rounded after repeated shaves such that there is an increase in the tip radius and a generally perceived decrease in shaving performance.

## SUMMARY OF THE INVENTION

In one aspect, the invention features, in general, a razor blade including a substrate with a cutting edge defined by a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of a chromium containing material on the layer of hard coating, and an outer layer of polytetrafluoroethylene coating on the overcoat layer.

In another aspect the invention features, in general, a shaving razor including a handle and a razor head with a blade having a substrate with a cutting edge defined by a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of a chromium containing material on the layer of hard coating, and an outer layer of polytetrafluoroethylene coating on the overcoat layer.

Particular embodiments of the invention may include one or more of the following features. In particular embodiments, the hard coating material can be made of carbon containing materials (e.g., diamond, amorphous diamond or DLC), nitrides, carbides, oxides or other ceramics. The hard coating layer can have a thickness less than 2,000 angstroms. The overcoat layer can be made of chromium or a chromium containing alloy compatible with polytetrafluoroethylene such as a chromium platinum alloy. The overcoat layer can be between 100 and 500 angstroms thick. The blade can include an interlayer between the substrate and the layer of hard coating. The interlayer can include niobium or a chromium containing material. The polytetrafluoroethylene can be Krytox LW1200 available from DuPont. The PTFE outer layer can be between 100 and 5000 angstroms thick.

In another aspect, the invention features, in general, making a razor blade by providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets, adding a layer of hard coating on the cutting edge, adding an overcoat layer of a chromium containing material on the layer of hard coating, and adding an outer layer of polytetrafluoroethylene coating over the overcoat layer.

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Particular embodiments of the invention may include one or more of the following features. In particular embodiments the layers can be added by physical vapor deposition (i.e., sputtering) or by chemical vapor deposition. The chromium containing layer, preferably chromium, can be sputter deposited under conditions that result in a compressively stressed coating. The sputter deposition of chromium containing materials can include applying a DC bias to the target that is more negative than –50 volts, preferably more negative than –200 volts. Alternatively an appropriate RF bias scheme can be used to achieve an equivalent chromium layer.

Embodiments of the invention may include one or more of the following advantages. The use of a chromium containing overcoat layer provides improved adhesion of the polytetrafluoroethylene outer layer to the hard coating layer. The razor blade has improved edge strength provided by hard coating and has reduced tip rounding with repeated shaves. Reduced tip rounding minimizes the increase in cutting force thereby maintaining excellent shaving performance. The razor blade has excellent shaving characteristics from the first shave onwards.

Other features and advantages of the invention will be apparent from the following description of a particular embodiment and from the claims.

## DESCRIPTION OF THE DRAWINGS

FIG. 1 is a vertical sectional view of a cutting edge portion of a razor blade.

FIG. 2 is a perspective view of a shaving razor including the FIG. 1 razor blade.

## DESCRIPTION OF A PARTICULAR EMBODIMENT

Referring to FIG. 1, there is shown razor blade 10 including substrate 12, interlayer 14, hard coating layer 16, overcoat layer 18, and outer layer 20. The substrate 12 is typically made of stainless steel (though other substrates can be employed) and has an ultimate edge sharpened to a tip radius of less than 1,000 angstroms, preferably 200 to 300 angstroms, and has a profile with side facets 22 at an included angle of between 15 and 30 degrees, preferably about 19 degrees, measured at 40 microns from the tip.

Interlayer 14 is used to facilitate bonding of the hard coating layer to the substrate. Examples of suitable interlayer material are niobium and chromium containing material. A particular interlayer is made of niobium greater than 100 angstroms and preferably less than 500 angstroms thick. PCT 92/03330 describes use of a niobium interlayer.

Hard coating layer 16 provides improved strength, corrosion resistance and shaving ability and can be made from carbon containing materials (e.g., diamond, amorphous diamond or DLC), nitrides (e.g., boron nitride, niobium nitride or titanium nitride), carbides (e.g., silicon carbide), oxides (e.g., alumina, zirconia) or other ceramic materials. The carbon containing materials can be doped with other elements, such as tungsten, titanium or chromium by including these additives, for example in the target during application by sputtering. The materials can also incorporate hydrogen, e.g., hydrogenated DLC. Preferably coating layer 16 is made of diamond, amorphous diamond or DLC. A particular embodiment includes DLC less than 2,000 angstroms, preferably less than 1,000 angstroms. DLC layers and methods of deposition are described in U.S. Pat. No. 5,232,568. As described in the "Handbook of Physical Vapor



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Deposition (PVD) Processing," DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond.

Overcoat layer 18 is used to reduce the tip rounding of the hard coated edge and to facilitate bonding of the outer layer to the hard coating while still maintaining the benefits of both. Overcoat layer 18 is preferably made of chromium containing material, e.g., chromium or chromium alloys that are compatible with polytetrafluoroethylene, e.g., CrPt. A particular overcoat layer is chromium about 100–200 angstroms thick. Blade 10 has a cutting edge that has less rounding with repeated shaves than it would have without the overcoat layer.

Outer layer 20 is used to provide reduced friction and includes polytetrafluoroethylene and is sometimes referred to as a telomer. A particular polytetrafluoroethylene material is Krytox LW 1200 available from DuPont. This material is a nonflammable and stable dry lubricant that consists of small particles that yield stable dispersions. It is furnished as an aqueous dispersion of 20% solids by weight and can be applied by dipping, spraying, or brushing, and can thereafter be air dried or melt coated. The layer is preferably less than 5,000 angstroms and could typically be 1,500 angstroms to 4,000 angstroms, and can be as thin as 100 angstroms, provided that a continuous coating is maintained. Provided that a continuous coating is achieved, reduced telomer coating thickness can provide improved first shave results. U.S. Pat. Nos. 5,263,256 and 5,985,459, which are hereby incorporated by reference, describe techniques which can be used to reduce the thickness of an applied telomer layer.

Razor blade 10 is made generally according to the processes described in the above referenced patents. A particular embodiment includes a niobium interlayer 14, DLC hard coating layer 16, chromium overcoat layer 18, and Krytox LW1200 polytetrafluoroethylene outer coat layer 20. Chromium overcoat layer 18 is deposited to a minimum of 100 angstroms and a maximum of 500 angstroms. It is deposited by sputtering using a DC bias (more negative than –50 volts and preferably more negative than –200 volts) and pressure of about 2 millitorr argon. The increased negative bias is believed to promote a compressive stress (as opposed to a tensile stress), in the chromium overcoat layer which is believed to promote improved resistance to tip rounding while maintaining good shaving performance. Blade 10 preferably has a tip radius of about 200–400 angstroms, measured by SEM after application of overcoat layer 18 and before adding outer layer 20.

Referring to FIG. 2, blade 10 can be used in shaving razor 110, which includes handle 112 and replaceable shaving cartridge 114. Cartridge 114 includes housing 116, which carries three blades 10, guard 120 and cap 122. Blades 10 are movably mounted, as described, e.g., in U.S. Pat. No. 5,918,369, which is incorporated by reference. Cartridge 114 also includes an interconnect member on which housing 116 is pivotally mounted at two arms 128. The interconnect member includes a base 127 which is replaceably connected to handle 112. Alternatively, blade 10 can be used in other razors having one, two or more than three blades, double-sided blades, and razors that do not have movable blades or pivoting heads where the cartridge is either replaceable or permanently attached to a razor handle.

In use, razor blade 10 has excellent shaving characteristics from the first shave onwards. Blade 10 has improved edge strength provided by hard coating and has reduced tip rounding with repeated shaves provided by the overlayer coating while maintaining excellent shave characteristics.

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Other embodiments of the invention are within the scope of the appended claims.

What is claimed is:

1. A razor blade comprising a substrate with a cutting edge defined by a sharpened tip and adjacent facets,
  - a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,
  - an overcoat layer of a chromium containing material on said layer of hard coating, and
  - an outer layer of polytetrafluoroethylene coating over said overcoat layer.
2. The blade of claim 1 wherein said hard carbon coating comprises diamond-like carbon material.
3. The blade of claim 2 wherein said overcoat layer consists of chromium.
4. The blade of claim 3 wherein said polytetrafluoroethylene is Krytox LW1200.
5. The blade of claim 2 further comprising a niobium interlayer between said substrate and said hard coating.
6. The blade of claim 1 wherein said hard carbon coating comprises amorphous diamond material.
7. The blade of claim 1 wherein said overcoat layer consists of chromium.
8. The blade of claim 1 wherein said overcoat layer consists of a chromium containing alloy compatible with polytetrafluoroethylene.
9. The blade of claim 8 wherein said alloy is a chromium platinum alloy.
10. The blade of claim 7, 8, 3, or 9 wherein said overcoat layer is compressively stressed.
11. The blade of claim 1 further comprising an interlayer between said substrate and said layer of hard coating.
12. The blade of claim 11 wherein said interlayer comprises niobium.
13. The blade of claim 11 wherein said interlayer comprises a chromium containing material.
14. The blade of claim 1 wherein said polytetrafluoroethylene is Krytox LW1200.
15. The blade of claim 1 wherein said hard coating layer has a thickness less than 2,000 angstroms.
16. The blade of claim 1 wherein said overcoat layer is between 100 and 500 angstroms thick.
17. The blade of claim 1 wherein said outer layer is between 100 and 5,000 angstroms thick.
18. The blade of claim 1, 3, 4 or 15 wherein said cutting edge has less rounding with repeated shaves than it would have without said overcoat layer.
19. The blade of claim 1 wherein said hard coating is doped with another element.
20. A shaving razor comprising
  - a handle,
  - a housing connected to said handle, and
  - at least one razor blade mounted in said housing, said blade comprising
    - a substrate with a cutting edge defined by a sharpened tip and adjacent facets,
    - a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,
    - an overcoat layer of a chromium containing material on said layer of hard coating, and
    - an outer layer of polytetrafluoroethylene coating over said overcoat layer.
21. The razor of claim 20 further comprising a niobium interlayer between said substrate and said hard coating.



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22. The razor of claim 20 wherein said overcoat layer consists of chromium.

23. The razor of claim 20 wherein said hard coating is doped with another element.

24. A method of making a razor blade comprising  
providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets,

adding a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,

adding an overcoat layer of a chromium containing material on said layer of hard coating, and

adding an outer layer of polytetrafluoroethylene coating over said overcoat layer.

25. The method of claim 24 wherein said adding a layer of hard coating includes vapor depositing a carbon containing material.

26. The method of claim 24 wherein said adding a layer of chromium containing material includes vapor depositing said chromium containing material.

27. The method of claim 26 wherein said adding a layer of chromium containing material includes sputter depositing under conditions to result in compressively stressed material.

28. A razor blade comprising

a substrate with a cutting edge defined by a sharpened tip and adjacent facets;

a layer of a hard carbon containing material, doped with another element, on the cutting edge;

an overcoat layer of a chromium containing material on the layer of the hard carbon containing material; and  
an outer layer of polytetrafluoroethylene over the overcoat layer.

29. The razor blade of claim 28, wherein the element is a metal.

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30. The razor blade of claim 29, wherein the metal is selected from the group consisting of tungsten and titanium.

31. The razor blade of claim 29, wherein the metal is chromium.

32. The razor blade of claim 31, wherein the hard carbon containing material is diamond-like carbon.

33. The razor blade of claim 28, wherein the hard carbon containing material is selected from the group consisting of diamond-like carbon and amorphous diamond.

34. The razor blade of claim 28, wherein the layer of hard carbon material has a thickness less than 2,000 angstroms, the overcoat layer has a thickness between 100 and 500 angstroms, and the outer layer has a thickness between 100 and 5,000 angstroms.

35. A shaving razor comprising

a handle,

a housing connected to the handle, and

at least one razor blade within the housing, the razor blade comprising

a substrate with a cutting edge defined by a sharpened tip and adjacent facets;

a layer of a hard carbon containing material, doped with another element, on the cutting edge;

an overcoat layer of a chromium containing material on the layer of the hard carbon containing material; and  
an outer layer of polytetrafluoroethylene over the overcoat layer.

36. The shaving razor of claim 35, wherein the element is a metal.

37. The shaving razor of claim 35, wherein the metal is chromium.

38. The shaving razor of claim 35, wherein the hard carbon containing material is selected from the group consisting of diamond-like carbon and amorphous diamond.

\* \* \* \* \*

UNITED STATES PATENT AND TRADEMARK OFFICE  
**CERTIFICATE OF CORRECTION**

PATENT NO. : 6,684,513 B1  
DATED : February 3, 2004  
INVENTOR(S) : Colin Clipstone et al.

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Title page.

Item [56], **References Cited**, U.S. PATENT DOCUMENTS, please replace "10/1975" and insert -- 11/1975 --

Column 1.

Line 10, delete "-" after "carbon"

Column 2.

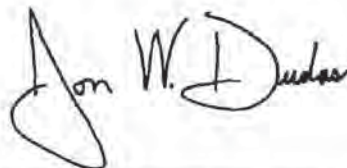
Line 2, insert -- , -- after "embodiments"

Column 6.

Line 37, delete "claim 35" and insert -- Claim 36 --

Signed and Sealed this

Ninth Day of November, 2004

A handwritten signature in black ink, reading "Jon W. Dudas". The signature is stylized, with a large loop for the "J" and a cursive "Dudas".

JON W. DUDAS  
*Director of the United States Patent and Trademark Office*

# EXHIBIT C







10-712 U.S. PTO  
09/515421

02/29/00

030	346.54	Class	Subclass	ISSUE CLASSIFICATION
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RB

PATENT NUMBER

6684513



6684513

## U.S. UTILITY Patent Application

HA	Q.I.P.E.	PATENT DATE
SCANNED	OK	FEB 03 2004

APPLICATION NO. 09/515421	CONT/PRIOR	CLASS 030	SUBCLASS 346.54	ART UNIT 3724	EXAMINER Payer
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APPLICANTS  
Colin Clipstone  
Steve Hahn  
Neville Sonnenberg  
Charles White

## Certificate

NOV 09 2004

## of Correction

TITLE  
Razor Blade technologyPTO-2010  
12/99

## ISSUING CLASSIFICATION

ORIGINAL		CROSS REFERENCE(S)					
CLASS	SUBCLASS	CLASS	SUBCLASS (ONE SUBCLASS PER BLOCK)				
030	346.54	030	346.53				
INTERNATIONAL CLASSIFICATION							
B26B	21/58						
B26B	21/60						

☐ Continued on Issue Slip Inside File Jacket

<input type="checkbox"/> <b>TERMINAL DISCLAIMER</b>	<b>DRAWINGS</b>			<b>CLAIMS ALLOWED</b>	
	Sheets Drwg. 1	Figs. Drwg. 2	Print Fig. 1	Total Claims 38	Print Claim for O.G. 20
<input type="checkbox"/> The term of this patent subsequent to _____ (date) has been disclaimed.	_____ (Assistant Examiner)			_____ (Date)	
<input type="checkbox"/> The term of this patent shall not extend beyond the expiration date of U.S. Patent No. _____	Hwei-Siu Payer Primary Examiner			9/24/03	
	_____ (Primary Examiner)			9/23/03 (Date)	
<input type="checkbox"/> The terminal _____ months of this patent have been disclaimed.	_____ (Legal Instruments Examiner)			_____ (Date)	
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Bib Data Sheet

<b>SERIAL NUMBER</b> 09/515,421	<b>FILING DATE</b> 02/29/2000 <b>RULE</b> -	<b>CLASS</b> 030	<b>GROUP ART UNIT</b> 3724	<b>ATTORNEY DOCKET NO.</b> 00216-483001	
<b>APPLICANTS</b> Colin John Clipstone, Weston, MA ; Steve Hahn, Wellesley, MA ; Neville Sonnenberg, Newton, MA ; Charles White, Lynnfield, MA ; Andrew Zhuk, Acton, MA ;					
<b>** CONTINUING DATA *****</b> <i>no</i>					
<b>** FOREIGN APPLICATIONS *****</b> <i>no</i>					
<b>IF REQUIRED, FOREIGN FILING LICENSE GRANTED ** 04/27/2000</b> -					
Foreign Priority claimed <input type="checkbox"/> yes <input checked="" type="checkbox"/> no 35 USC 119 (a-d) conditions met <input type="checkbox"/> yes <input checked="" type="checkbox"/> no <input type="checkbox"/> Met after Allowance <i>1/7</i> Verified and Acknowledged _____ Examiner's Signature _____ Initials _____		<b>STATE OR COUNTRY</b> MA	<b>SHEETS DRAWING</b> 1	<b>TOTAL CLAIMS</b> 34	<b>INDEPENDENT CLAIMS</b> 3
<b>ADDRESS</b> William E Booth Fish & Richardson PC 255 Franklin Street Boston, MA 02110-2804					
<b>TITLE</b> Razor Blade technology					
<b>FILING FEE RECEIVED</b> 1386	FEES: Authority has been given in Paper No. _____ to charge/credit DEPOSIT ACCOUNT No. _____ for following:		<input type="checkbox"/> All Fees <input type="checkbox"/> 1.16 Fees ( Filing ) <input type="checkbox"/> 1.17 Fees ( Processing Ext. of time ) <input type="checkbox"/> 1.18 Fees ( Issue ) <input type="checkbox"/> Other _____ <input type="checkbox"/> Credit		

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Date January 4, 2002

To H. Payer  
U.S. Patent and Trademark Office (Patent)  
Assistant Commissioner for Patents  
Washington, DC 20231  
Telephone: (703) 308-1405

Facsimile number 00216-48300001 / 703-872-9302

From William E. Booth

Re RAZOR BLADE TECHNOLOGY  
Application No.: 09/515,421  
Our Ref.: 00216-483001

Number of pages  
Including this page 15

Message Response to office action previously filed October 19, 2001.

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PATENT  
ATTORNEY DOCKET NO. 00216-483001

The Patent and Trademark Office date stamp sets forth the date of receipt of:

Applicant or Patentee Neville Sonnenberg et al.

No. (Application, Appeal, Interference, Patent, Reexam) 09/515,421

Filing or Issue Date February 29, 2000

Title: RAZOR BLADE TECHNOLOGY

☐ Transmittal Letter (2 Copies) ☐ With Pet. for Ext.  
☐ Assignment 9 Pages ☐ Status Inquiry  
☒ Amendment/Response 9 Pages ☐ Declaration  
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☐ Notice of Missing Parts  
☐ Combined Declaration and Power of Attorney  
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☒ Other Retracting Reference (3 pages)

Atty/Sec. RCN Client/  
Initials MB/jq Matter Name Gillette/Case 8073 Date 10/19/01

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Attorney's Docket No.: 00216-483001 / Case 8073



APPLICATION  
FOR  
UNITED STATES LETTERS PATENT

TITLE: RAZOR BLADE TECHNOLOGY

APPLICANT: NEVILLE SONNENBERG, ANDREW ZHUK, CHARLES  
WHITE, STEVEN HAHN AND COLIN CLIPSTONE

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Date of Deposit February 29, 2000

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Samantha Bell  
Typed or Printed Name of Person Signing Certificate

GILLETTE-DSC-0220511

Attorney Docket No. 00216-483001

### Razor Blade Technology

The invention relates to improvements to razors and razor blades.

A razor blade is typically formed of a suitable substrate material such as stainless steel, and a cutting edge is formed with a wedge-shaped configuration with an ultimate tip having a radius less than about 1000 angstroms, e.g., about 200 - 300 angstroms. Hard coatings such as diamond, amorphous diamond, diamond-like carbon (DLC) material, nitrides, carbides, oxides or ceramics are often used to improve strength, corrosion resistance and shaving ability, maintaining needed strength while permitting thinner edges with lower cutting forces to be used. Polytetrafluoroethylene (PTFE) outer layer can be used to provide friction reduction. Interlayers of niobium or chromium containing materials can aid in improving the binding between the substrate, typically stainless steel, and hard carbon coatings, such as DLC. Examples of razor blade cutting edge structures and processes of manufacture are described in U.S. Patents Nos. 5,295,305; 5,232,568; 4,933,058; 5,032,243; 5,497,550; 5,940,975; 5,669,144; EP 0591334<sup>9</sup>, and PCT 92/03330, which are hereby incorporated by reference.

In use, the ultimate tip of the edges having hard coatings and polytetrafluoroethylene outer layers can become more rounded after repeated shaves such that there is an increase in the tip radius and a generally perceived decrease in shaving performance.

### Summary of the Invention

In one aspect, the invention features, in general, a razor blade including a substrate with a cutting edge defined by a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of a chromium containing material on the layer of hard coating, and an outer layer of polytetrafluoroethylene coating on the overcoat layer.

In another aspect the invention features, in general, a shaving razor including a handle and a razor head with a blade having a substrate with a cutting edge defined by a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of a chromium containing material on the layer of hard coating, and an outer layer of polytetrafluoroethylene



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coating on the overcoat layer.

Particular embodiments of the invention may include one or more of the following features. In particular embodiments, the hard coating material can be made of carbon containing materials (e.g., diamond, amorphous diamond or DLC), nitrides, carbides, oxides or other ceramics. The hard coating layer can have a thickness less than 2,000 angstroms. The overcoat layer can be made of chromium or a chromium containing alloy compatible with polytetrafluoroethylene such as a chromium platinum alloy. The overcoat layer can be between 100 and 500 angstroms thick. The blade can include an interlayer between the substrate and the layer of hard coating. The interlayer can include niobium or a chromium containing material. The polytetrafluoroethylene can be Krytox LW1200 available from DuPont. The PTFE outer layer can be between 100 and 5000 angstroms thick.

In another aspect, the invention features, in general, making a razor blade by providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets, adding a layer of hard coating on the cutting edge, adding an overcoat layer of a chromium containing material on the layer of hard coating, and adding an outer layer of polytetrafluoroethylene coating over the overcoat layer.

Particular embodiments of the invention may include one or more of the following features. In particular embodiments the layers can be added by physical vapor deposition (i.e., sputtering) or by chemical vapor deposition. The chromium containing layer, preferably chromium, can be sputter deposited under conditions that result in a compressively stressed coating. The sputter deposition of chromium containing materials can include applying a DC bias to the target that is more negative than -50 volts, preferably more negative than -200 volts. Alternatively an appropriate RF bias scheme can be used to achieve an equivalent chromium layer.

Embodiments of the invention may include one or more of the following advantages. The use of a chromium containing overcoat layer provides improved adhesion of the polytetrafluorethylene outer layer to the hard coating layer. The razor blade has improved edge strength provided by hard coating and has reduced tip rounding with repeated shaves. Reduced

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tip rounding minimizes the increase in cutting force thereby maintaining excellent shaving performance. The razor blade has excellent shaving characteristics from the first shave onwards.

Other features and advantages of the invention will be apparent from the following description of a particular embodiment and from the claims.

5

#### Description of the Drawings

FIG. 1 is a vertical sectional view of a cutting edge portion of a razor blade.

FIG.2 is a perspective view of a shaving razor including the FIG. 1 razor blade.

#### Description of a Particular Embodiment

Referring to FIG. 1, there is shown razor blade 10 including substrate 12, interlayer 14, hard coating layer 16, overcoat layer 18, and outer layer 20. The substrate 12 is typically made of stainless steel (though other substrates can be employed) and has an ultimate edge sharpened to a tip radius of less than 1,000 angstroms, preferably 200 to 300 angstroms, and has a profile with side facets 22 at an included angle of between 15 and 30 degrees, preferably about 19 degrees, measured at 40 microns from the tip.

Interlayer 14 is used to facilitate bonding of the hard coating layer to the substrate. Examples of suitable interlayer material are niobium and chromium containing material. A particular interlayer is made of niobium greater than 100 angstroms and preferably less than 500 angstroms thick. PCT 92/03330 describes use of a niobium interlayer.

Hard coating layer 16 provides improved strength, corrosion resistance and shaving ability and can be made from carbon containing materials (e.g., diamond, amorphous diamond or DLC), nitrides (e.g., boron nitride, niobium nitride or titanium nitride), carbides (e.g., silicon carbide), oxides (e.g., alumina, zirconia) or other ceramic materials. The carbon containing materials can be doped with other elements, such as tungsten, titanium or chromium by including these additives, for example in the target during application by sputtering. The materials can also incorporate hydrogen, e.g., hydrogenated DLC. Preferably coating layer 16 is made of diamond, amorphous diamond or DLC. A particular embodiment includes DLC less than 2,000



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angstroms, preferably less than 1,000 angstroms. DLC layers and methods of deposition are described in U.S. Patent No. 5,232,568. As described in the "Handbook of Physical Vapor Deposition (PVD) Processing," DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond.

5 Overcoat layer 18 is used to reduce the tip rounding of the hard coated edge and to facilitate bonding of the outer layer to the hard coating while still maintaining the benefits of both. Overcoat layer 18 is preferably made of chromium containing material, e.g., chromium or chromium alloys that are compatible with polytetrafluoroethylene, e.g., CrPt. A particular overcoat layer is chromium about 100-200 angstroms thick. Blade 10 has a cutting edge that has  
10 less rounding with repeated shaves than it would have without the overcoat layer.

Outer layer 20 is used to provide reduced friction and includes polytetrafluoroethylene and is sometimes referred to as a telomer. A particular polytetrafluoroethylene material is Krytox LW 1200 available from DuPont. This material is a nonflammable and stable dry lubricant that consists of small particles that yield stable dispersions. It is furnished as an aqueous dispersion of 20% solids by weight and can be applied by dipping, spraying, or brushing, and can thereafter be air dried or melt coated. The layer is preferably less than 5,000 angstroms and could typically be 1,500 angstroms to 4,000 angstroms, and can be as thin as 100 angstroms, provided that a continuous coating is maintained. Provided that a continuous coating is achieved, reduced telomer coating thickness can provide improved first shave results. U.S. Patents Nos.  
15 5,263,256 and 5,985,459, which are hereby incorporated by reference, describe techniques which can be used to reduce the thickness of an applied telomer layer.  
20

Razor blade 10 is made generally according to the processes described in the above referenced patents. A particular embodiment includes a niobium interlayer 14, DLC hard coating layer 16, chromium overcoat layer 18, and Krytox LW1200 polytetrafluoroethylene outer coat  
25 layer 20. Chromium overcoat layer 18 is deposited to a minimum of 100 angstroms and a maximum of 500 angstroms. It is deposited by sputtering using a DC bias (more negative than -50 volts and preferably more negative than -200 volts) and pressure of about 2 millitorr argon. The increased negative bias is believed to promote a compressive stress (as opposed to a tensile

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stress), in the chromium overcoat layer which is believed to promote improved resistance to tip rounding while maintaining good shaving performance. Blade 10 preferably has a tip radius of about 200- 400 angstroms, measured by SEM after application of overcoat layer 18 and before adding outer layer 20.

Referring to FIG. 2, blade 10 can be used in shaving razor 110, which includes handle 112 and replaceable shaving cartridge 114. Cartridge 14 includes housing 116, which carries three blades 10, guard 120 and cap 122. Blades 10 are movably mounted, as described, e.g., in U.S. Patent No. 5,918,369, which is incorporated by reference. Cartridge 114 also includes interconnect member 124 on which housing 116 is pivotally mounted at two arms 128.

Interconnect member 124 includes a base 127 which is replaceably connected to handle 112. Alternatively, blade 10 can be used in other razors having one, two or more than three blades, double-sided blades, and razors that do not have movable blades or pivoting heads where the cartridge is either replaceable or permanently attached to a razor handle.

In use, razor blade 10 has excellent shaving characteristics from the first shave onwards. Blade 10 has improved edge strength provided by hard coating and has reduced tip rounding with repeated shaves provided by the overlayer coating while maintaining excellent shave characteristics.

Other embodiments of the invention are within the scope of the appended claims.

What is claimed is:



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Sub  
1. A razor blade comprising

2 a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
3 a layer of hard coating on said cutting edge,

4 an overcoat layer of a chromium containing material on said layer of hard coating, and  
5 an outer layer of polytetrafluoroethylene coating over said overcoat layer.

1 2. The blade of claim 1 wherein said hard coating is made of a carbon containing material.

1 3. The blade of claim 2 wherein said carbon containing material comprises diamond.

2 4. The blade of claim 2 wherein said hard carbon coating comprises diamond-like carbon  
material.

2 5. The blade of claim 2 wherein said hard carbon coating comprises amorphous diamond  
material.

6. The blade of claim 1 wherein said overcoat layer consists of chromium.

2 7. The blade of claim 1 wherein said overcoat layer consists of a chromium containing alloy  
compatible with polytetrafluoroethylene.

1 8. The blade of claim 4 wherein said overcoat layer consists of chromium.

1 9. The blade of claim 7 wherein said alloy is a chromium platinum alloy.

Sub  
10. The blade of claim 1 further comprising an interlayer between said substrate and said layer of



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2 ~~hard carbon coating.~~

1 11. The blade of claim 10 wherein said interlayer comprises niobium.

1 12. The blade of claim 10 wherein said interlayer comprises a chromium containing material.

1 13. The blade of claim 6, 7, 8, or 9 wherein said overcoat layer is compressively stressed.

1 14. The blade of claim 1 wherein said polytetrafluoroethylene is Krytox LW1200.

1 15. The blade of claim 4 further comprising a niobium interlayer between said substrate and said  
2 hard coating.

3 16. The blade of claim 8 wherein said polytetrafluoroethylene is Krytox LW1200.

4 17. The blade of claim 1 wherein said hard coating layer has a thickness less than 2,000  
5 angstroms.

6 18. The blade of claim 1 wherein said overcoat layer is between 100 and 500 angstroms thick.

7 19. The blade of claim 1 wherein said outer layer is between 100 and 5,000 angstroms thick.

8 20. The blade of claim 1, 8, 16 or 17 wherein said blade has a cutting edge that has less rounding  
9 with repeated shaves than it would have without said overcoat layer.

10 21. A shaving razor comprising  
11 a handle,  
12 a housing connected to said handle, and  
13



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4 at least one razor blade mounted in said housing, said blade comprising  
5 a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
6 a layer of hard coating on said cutting edge,  
7 an overcoat layer of a chromium containing material on said layer of hard coating, and  
8 a outer layer of polytetrafluoroethylene coating over said overcoat layer.

1 22. The razor of claim 21 wherein said hard coating is made of a carbon containing material.

1 23. The razor of claim 22 further comprising a niobium interlayer between said substrate and  
2 said hard coating.

1 24. The razor of claim 21 or 22 wherein said overcoat layer consists of chromium.

1 25. A method of making a razor blade comprising  
2 providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
3 adding a layer of hard coating on said cutting edge,  
4 adding an overcoat layer of a chromium containing material on said layer of hard coating, and  
5 adding an outer layer of polytetrafluoroethylene coating over said overcoat layer.

1 26. The method of claim 25 wherein said adding a layer of hard coating includes vapor  
2 depositing a carbon containing material.

1 27. The method of claim 25 wherein said adding a layer of chromium containing material  
2 includes vapor depositing said chromium containing material.

1 28. The method of claim 27 wherein said adding a layer of chromium containing material  
2 includes sputter depositing under conditions to result in compressively stressed material.

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1 29. The method of claim 28 wherein said sputtering includes applying a DC bias to said target  
2 that is more negative than -50 volts or an equivalent RF bias scheme.

1 30. The method of claim 28 wherein said sputtering includes applying a DC bias to said target  
2 that is more negative than -200 volts or an equivalent RF bias scheme.

00216-483001

[illegible]

## 10

*[Signature]*



<b>Office Action Summary</b>	Application No.	Applicant(s)	
	09/515,421	CLIPSTONE ET AL.	
	Examiner	Art Unit	
	Hwei-Siu Payer	3724	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136 (a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

1) ☐ Responsive to communication(s) filed on \_\_\_\_.

2a) ☐ This action is **FINAL**.                      2b) ☒ This action is non-final.

3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

4) ☒ Claim(s) 1-30 is/are pending in the application.

4a) Of the above claim(s) \_\_\_\_ is/are withdrawn from consideration.

5) ☐ Claim(s) \_\_\_\_ is/are allowed.

6) ☒ Claim(s) 1-30 is/are rejected.

7) ☐ Claim(s) \_\_\_\_ is/are objected to.

8) ☐ Claims \_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

9) ☒ The specification is objected to by the Examiner.

10) ☐ The drawing(s) filed on \_\_\_\_ is/are objected to by the Examiner.

11) ☐ The proposed drawing correction filed on \_\_\_\_ is: a) ☐ approved b) ☐ disapproved.

12) ☐ The oath or declaration is objected to by the Examiner.

**Priority under 35 U.S.C. § 119**

13) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

a) ☐ All   b) ☐ Some \* c) ☐ None of:

1. ☐ Certified copies of the priority documents have been received.

2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_.

3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

14) ☐ Acknowledgement is made of a claim for domestic priority under 35 U.S.C. § 119(e).

**Attachment(s)**

15) ☒ Notice of References Cited (PTO-892)

16) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)

17) ☒ Information Disclosure Statement(s) (PTO-1449) Paper No(s) 4.

18) ☐ Interview Summary (PTO-413) Paper No(s). \_\_\_\_

19) ☐ Notice of Informal Patent Application (PTO-152)

20) ☐ Other:

Application/Control Number: 09/515,421

Page 2

Art Unit: 3724

## **Detailed Action**

### **Objection to the Specification**

The disclosure is objected to because of the following informalities: On page 5, reference numeral "124" is not found in any drawings.

Appropriate correction is required.

### **Claims Rejection - 35 U.S.C. 112, second paragraph**

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

2. Claims 10-12, 29 and 30 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

(1) In claim 10, "said layer of hard carbon coating" has no clear antecedent basis. The phrase should read —said layer of hard coating—.

(2) In claims 29 and 30, "said target" lacks antecedent basis.

### **Claims Rejection – 35 U.S.C. 102(b)**

1. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –



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(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

2. Claims 1, 6-9, 17-20, 25 and 27 are rejected under 35 U.S.C. 102(b) as being clearly anticipated by Lane et al. '579

**Claims Rejection - 35 U.S.C. 103(a)**

1. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

2. Claims 2-4, 10, 11 and 15 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 in view of Hahn et al. '305.

Lane et al. shows the claimed invention except it does not specifically mention the hard coating (I) being made of a carbon containing material such as diamond or diamond-like carbon material, and it lacks a niobium interlayer disposed between the substrate (101) and the hard coating (I).

Hahn et al. shows (Fig.3) a hard coating such as diamond or diamond-like carbon material (60) for hardening the cutting edge of a razor blade and a niobium interlayer (58) disposed between the substrate (50) and the hard coating (60) for improving adhesion of the hard coating (60).

It would have been obvious to one skilled in the art to select a well known hard coating material such as the diamond or diamond-like carbon material of <sup>Hahn</sup>Parent et al.

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for the hard coating material of Lane et al. The modification is obvious since it would only involve selecting one known type of hard coating material for another for hardening the cutting edge of the razor blade. Also, It would have been obvious to one skilled in the art to further modify Lane et al. by providing the razor blade with a niobium interlayer for increasing the adhesion between the substrate and the hard coating as taught by Hahn et al.

3. Claims 2, 5 and 26 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 in view of Decker et al. '549.

Lane et al. shows the claimed invention except it does not specifically mention the hard coating (I) being made of a carbon containing material such as amorphous diamond.

Decker et al. shows (Fig.3) a hard coating such as amorphous diamond material (60) for hardening the cutting edge of a razor blade.

It would have been obvious to one skilled in the art to select a well known hard coating material such as the amorphous diamond material of Decker et al. for the hard coating material of Lane et al. The modification is obvious since it would only involve selecting one known type of hard coating material for another for hardening the cutting edge of a razor blade.

4. Claims 10 and 12 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 in view of Bache et al. '243.

Lane et al. shows the claimed invention except it lacks a chromium interlayer disposed between the substrate (101) and the hard coating (I).



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Art Unit: 3724

Bache et al. shows a chromium interlayer (see column 7, lines 53-55) disposed between a substrate and a hard coating (60) for improving adhesion of the hard coating (60).

It would have been obvious to one skilled in the art to modify Lane et al. by providing the razor blade with a chromium interlayer for increasing the adhesion between the substrate and the hard coating as taught by Bache et al.

5. Claims 13 and 28-30 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 in view of Parent et al. '568.

Lane et al. shows the claimed invention except for the claimed sputter depositing condition.

Parent et al. shows sputter depositing a coating onto a razor blade including applying a DC bias to a sputter target that is more negative than -200 volts (see column 5, lines 17-12) to achieve a desired deposited coating on the razor blade.

It would have been obvious to one skilled in the art to modify Lane et al. by selecting a well known sputter depositing condition of Parent et al. for that of Lane et al. The modification is obvious since it would only involve substituting one known condition for another for sputter depositing a coating on a razor blade.

6. Claims 14 and 16 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579.

Lane et al. shows the claimed invention except it does not specifically mention the polytetrafluoroethylene (III) being Krytox LW 1200. To select a commercially

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available polytetrafluoroethylene such as Kcytox LW 1200 from DuPont is deemed to be an obvious matter of preference and not patentably distinct.

7. Claims 21 and 24/21 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. in view of Apprille, Jr. et al. '369.

Lane et al. shows the claimed invention except it lacks a housing for mounting the blade (Fig.5) and a handle for supporting the housing.

Apprille, Jr. et al. shows a shaving razor (10) comprising a housing (16) for mounting a blade (18) and a handle (12) supporting the housing (16).

It would have been obvious to one skilled in the art to modify Lane et al. by providing a housing for mounting the blade and a handle for supporting the housing in order to facilitate manipulating the blade during shaving operation as taught by Apprille, Jr. et al.

8. Claims 22 and 24/22/21 are rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 and Apprille, Jr. et al. as in claim 21 and further in view of Decker et al. '549.

Lane et al. as modified shows the claimed invention except it does not specifically mention the hard coating (I) being made of a carbon containing material.

Decker et al. shows (Fig.3) a hard coating made of a carbon containing material.

It would have been obvious to one skilled in the art to select a well known hard coating material such as the carbon containing material Decker et al. for the hard coating material of Lane et al. The modification is obvious since it would only involve



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selecting one known type of hard coating material for another for hardening the cutting edge of a razor blade.

9. Claim 23 is rejected under 35 U.S.C. 103(a) as being unpatentable over Lane et al. '579 and Apprille, Jr. et al. as in claim 22 and further in view of Hahn et al. '305.

Lane et al. as modified shows the claimed invention except it lacks a niobium interlayer disposed between the substrate (101) and the hard coating (I).

Hahn et al. shows (Fig.3) a niobium interlayer (58) disposed between the substrate (50) and the hard coating (60) for improving adhesion of the hard coating (60).

It would have been obvious to one skilled in the art to modify Lane et al. by providing the razor blade with a niobium interlayer for increasing the adhesion between the substrate and the hard coating as taught by Hahn et al.

#### **Prior Art Citations**

The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. Kiss et al, Wilkinson, Blochl, Sanderson '551, Lane, Sanderson '703, Lindstrom et al. and Jones are cited as art of interest.

#### **Point of Contact**

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Hwei-Siu Payer whose telephone number is 703-308-1405. The examiner can normally be reached on Monday through Friday, 7:00 am to 4:00 pm.



Application/Control Number: 09/515,421

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Art Unit: 3724

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Rinaldi Rada can be reached on 703-308-2187. The fax phone numbers for the organization where this application or proceeding is assigned are 703-305-3579 for regular communications and 703-305-3579 for After Final communications.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-1148.

H Payer  
May 22, 2001



**Hwei-Siu Payer**  
**Primary Examiner**

<b>Notice of Reference Cited</b>	Application/Control No. 09/515,421	Applicant(s)/Patent Under Examination IPSTONE ET AL.	
	Examiner Hwei-Siu Payer	Art Unit 3724	Page 1 of 1

## U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification	
	A	US-3,345,202-A	10-1967	Kiss et al.	30	346.53
	B	US-3,480,483-A	11-1969	Wilkinson	30	346.53
	C	US-3,508,957-A	04-1970	Bloch	30	346.53
	D	US-3,743,551-A	07-1973	Sanderson	30	346.54
	E	US-3,754,329-A	08-1973	Lane	30	346.53
	F	US-3,774,703-A	11-1973	Sanderson	30	346.53
	G	US-3,837,896-A	09-1974	Lindstrom et al.	30	346.53
	H	US-3,890,109-A	06-1975	Jones	30	346.53
	I	US-3,911,579-A	11-1975	Lane et al.	30	346.53
	J	US- -				
	K	US- -				
	L	US- -				
	M	US- -				

## FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification	
	N	- -					
	O	- -					
	P	- -					
	Q	- -					
	R	- -					
	S	- -					
	T	- -					

## NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)					
	U						
	V						
	W						
	X						

\*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)  
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

U.S. Patent and Trademark Office  
PTO-892 (Rev. 01-2001)

Notice of References Cited

Part of Paper No. 5

GILLETTE-DSC-0220710





Attorney's Ltr. No.: 00216-483001 / Case 8073

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Neville Sonnenberg et al. Art Unit : 3724  
 Serial No. : 09/515,421 Examiner : Hwei-Siu Payer  
 Filed : February 29, 2000  
 Title : RAZOR BLADE TECHNOLOGY

Commissioner for Patents  
 Washington, D.C. 20231

# 8/ B W/ att  
 1/16/02  
 RECEIVED  
 JAN 14 2002  
 TC 3700 MAIL ROOM

RESPONSE

In response to the action mailed May 25, 2001, please amend the application as follows:

In the specification:

Please amend the paragraph beginning at page 5, line 5 to read as follows:

--Referring to FIG. 2, blade 10 can be used in shaving razor 110, which includes handle 112 and replaceable shaving cartridge 114. Cartridge 14 includes housing 116, which carries three blades 10, guard 120 and cap 122. Blades 10 are movably mounted, as described, e.g., in U.S. Patent No. 5,918,369, which is incorporated by reference. Cartridge 114 also includes an interconnect member on which housing 116 is pivotally mounted at two arms 128. The interconnect member includes a base 127 which is replaceably connected to handle 112. Alternatively, blade 10 can be used in other razors having one, two or more than three blades, double-sided blades, and razors that do not have movable blades or pivoting heads where the cartridge is either replaceable or permanently attached to a razor handle.--

In the claims:

Please cancel claim 3.

Please amend claims 1, 2, 21, 22, and 25 as follows:

CERTIFICATE OF MAILING BY FIRST CLASS MAIL

I hereby certify under 37 CFR §1.8(a) that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage on the date indicated below and is addressed to the Commissioner for Patents, Washington, D.C. 20231.

October 19, 2001  
 Date of Deposit

Signature *Jennifer Reveille*

Typed or Printed Name of Person Signing Certificate  
 Jennifer Reveille



Applicant : Neville Sonnabend et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 2

Attorney's Doc. No.: 00216-483001 / Case 8073

B2

1. (Amended) A razor blade comprising a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.

2. (Amended) The blade of claim 1 wherein said hard coating is made of amorphous carbon containing material.

Sub C3  
B3

21. (Amended) A shaving razor comprising  
a handle,  
a housing connected to said handle, and  
at least one razor blade mounted in said housing, said blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material,  
an overcoat layer of a chromium containing material on said layer of hard coating,  
and  
a outer layer of polytetrafluoroethylene coating over said overcoat layer.

22. (Amended) The razor of claim 21 wherein said hard coating is made of amorphous carbon containing material.

B4

25. (Amended) A method of making a razor blade comprising  
providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
adding a layer of hard coating on said cutting edge, said hard coating being made of amorphous material,  
adding an overcoat layer of a chromium containing material on said layer of hard coating,  
and

B

Applicant : Neville Sonnensberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 3

Attorney's De, et No.: 00216-483001 / Case 8073

B4 [ adding an outer layer of polytetrafluoroethylene coating over said overcoat layer.

---

B



Applicant : Neville Sonnetberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 4

Attorney's Docket No.: 00216-483001 / Case 8073

#### REMARKS

The invention, as claimed in independent claims 1, 21 and 25, is directed to a razor blade having a substrate with a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of chromium containing material thereon, and an outer layer of polytetrafluoroethylene (PTFE) thereon. The claims have been amended to recite that the hard coating is made of "amorphous material," and, so limited, exclude crystalline material. Such amorphous material includes "diamond-like carbon" ("DLC"), as recited in dependent claim 4, and "amorphous diamond material," as recited in dependent claim 5. As noted at page 4, lines 2-4, with reference to the "Handbook of Physical Vapor Deposition (PVD) Processing," "DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond."

In the claimed invention, the use of an amorphous hard coating is advantageous in obtaining the desired tip geometry. When depositing a crystalline hard coating, the crystals can grow to a size that is larger than the desired tip radius, frustrating efforts to obtain a desired coating thickness and tip radius. This problem does not exist when depositing amorphous material, making it easier to deposit the desired amorphous hard coating thickness and obtain the desired tip radius shape. Such amorphous materials have been used extensively with a telomer (PTFE) outer coating, as described in the cited Hahn patent. A problem that has been noted with at least some amorphous carbon hard coating materials used with PTFE (in particular amorphous DLC), however, is that there is substantial rounding of the blade tips with repeated use during wet shaving, resulting in an increase in required cutting force during shaving.

The inventors found that, by using a chromium containing layer between an amorphous hard coating material and a PTFE layer, they could maintain the good edge strength provided by hard coating and have reduced tip rounding with repeated shaves. The razor blade has excellent shaving characteristics from the first shave onwards. These advantages are described in the application at page 2, line 27 to page 3, line 2. This is an unexpected result for the amorphous hard coating material, because it would not be expected that a material that is softer than the hard



Applicant : Neville Sonnenberg et al.  
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The remaining claims depend on the independent claims and are allowable with them.

Attached is a marked-up version of the changes being made by the current amendment.

Applicant asks that all claims be allowed. Enclosed is a \$400 check for the Petition for Extension of Time fee. Please apply any other charges or credits to Deposit Account No. 06-1050.

Date: Oct 19, 2001

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Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 7

Attorney's Docket No.: 00216-483001 / Case 8073

**Version with markings to show changes made**

**In the specification:**

The paragraph beginning at page 5, line 5 has been amended as follows:

--Referring to FIG. 2, blade 10 can be used in shaving razor 110, which includes handle 112 and replaceable shaving cartridge 114. Cartridge 114 includes housing 116, which carries three blades 10, guard 120 and cap 122. Blades 10 are movably mounted, as described, e.g., in U.S. Patent No. 5,918,369, which is incorporated by reference. Cartridge 114 also includes an interconnect member [124] on which housing 116 is pivotally mounted at two arms 128. The interconnect [Interconnect] member [124] includes a base 127 which is replaceably connected to handle 112. Alternatively, blade 10 can be used in other razors having one, two or more than three blades, double-sided blades, and razors that do not have movable blades or pivoting heads where the cartridge is either replaceable or permanently attached to a razor handle.--

**In the claims:**

Claim 3 has been cancelled.

Claims 1, 2, 21, 22 and 25 have been amended as follows:

1. (Amended) A razor blade comprising  
substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.
2. (Amended) The blade of claim 1 wherein said hard coating is made of [a] amorphous carbon containing material.
21. (Amended) A shaving razor comprising  
a handle,



<b>Office Action Summary</b>	Application No. 09/515,421	Applicant(s) CLIPSTONE ET AL.	
	Examiner Hwei-Siu C. Payer	Art Unit 3724	

**-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --**

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

1) ☒ Responsive to communication(s) filed on 19 October 2001.

2a) ☐ This action is FINAL.                      2b) ☒ This action is non-final.

3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

4) ☒ Claim(s) 1, 2 and 4-30 is/are pending in the application.

4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.

5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.

6) ☒ Claim(s) 1, 2 and 4-30 is/are rejected.

7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.

8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

9) ☐ The specification is objected to by the Examiner.

10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

11) ☐ The proposed drawing correction filed on \_\_\_\_\_ is: a) ☐ approved b) ☐ disapproved by the Examiner.  
If approved, corrected drawings are required in reply to this Office action.

12) ☐ The oath or declaration is objected to by the Examiner.

**Priority under 35 U.S.C. §§ 119 and 120**

13) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).  
a) ☐ All b) ☐ Some \* c) ☐ None of:  
1. ☐ Certified copies of the priority documents have been received.  
2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.  
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).  
\* See the attached detailed Office action for a list of the certified copies not received.

14) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. § 119(e) (to a provisional application).  
a) ☐ The translation of the foreign language provisional application has been received.

15) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121.

**Attachment(s)**

1) <input type="checkbox"/> Notice of References Cited (PTO-892)	4) <input type="checkbox"/> Interview Summary (PTO-413) Paper No(s). _____
2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)	5) <input type="checkbox"/> Notice of Informal Patent Application (PTO-152)
3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO-1449) Paper No(s) <u>6</u>	6) <input type="checkbox"/> Other: _____



Application/Control Number: 09/515,421  
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Page 2

## Detailed Action

The amendment filed on 10-19-2001 has been entered.

### Claims Objection

Claims 21-24 are objected to because of the following informalities:

In claim 21, line 10, "a outer layer" should read --an outer layer--.

Appropriate correction is required.

### Claims Rejection - 35 U.S.C. 112, second paragraph

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

2. Claims 10-12, 20, 29 and 30 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

(1) In claim 10, lines 1-2, "said layer of hard carbon coating" has no clear antecedent basis. The phrase should read --said layer of hard coating--.

(2) In claim 20, "a cutting edge" is vague. Is it in addition to "a cutting edge" of claim 1? Furthermore, it is unclear exactly what structure of the blade is being claimed therein.

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Art Unit: 3724

Page 3

(3) In claims 29 and 30, "said target" has no antecedent basis. Further, "an equivalent RF bias scheme" is vague and indefinite.

**Claims Rejection - 35 U.S.C. 103(a)**

1. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

2. Claims 1, 2, 4, 6-11 and 13-30 are rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) in view of Lane et al. (U.S. Patent No. 3,911,579).

Hahn et al. discloses a razor blade (Fig.3) comprising a substrate (50) with a cutting edge defined by a sharpened tip (70) and adjacent facets (62,64); a layer of hard coating (60) on said cutting edge, the hard coating (60) being made of amorphous material such as diamond-like carbon material (see column 3, lines 65-66); a niobium interlayer (58) between the substrate (50) and the hard coating (60); and a polytetrafluoroethylene outer layer (72) substantially as claimed except Hahn et al. lacks an overcoat layer of chromium between the hard coating (60) and the polytetrafluoroethylene outer layer (72).

Lane et al. shows a razor blade comprising an overcoat layer (II) of a chromium containing material provided between a layer of hard coating (I) and an outer layer of



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polytetrafluoroethylene (III) by RF sputtering process or DC sputtering process (see column 6, lines 26-32). The chromium overcoat layer (II) provides a hard and durable shaving edge (see column 6, lines 11-22).

It would have been obvious to one skilled in the art to modify Hahn et al. by providing a layer of chromium coating between the hard coating (60) and the polytetrafluoroethylene layer to aid in facilitating a hard and durable shaving edge for the razor blade as taught by Lane et al.

It is noted claims 14 and 16 call for the polytetrafluoroethylene outer layer (72) being Krytox LW 1200. To select a commercially available polytetrafluoroethylene such as Krytox LW 1200 from DuPont is deemed to be an obvious matter of preference and not patentably distinct.

Claims 13 and 28-30 call for the sputter depositing condition (i.e. applying a DC bias of more negative than -200 volts) of the chromium overcoat layer. The claimed depositing condition is not patentably distinct over Hahn et al. as modified, since the exact sputtering condition depends more upon the final desired shape of the deposited coating on the razor blade than on any inventive concept.

3. Claim 5 is rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) and Lane et al. (U.S. Patent No. 3,911,579) as applied to claim 2 above, and further in view of Decker et al. (U.S. Patent No. 5,799,549).

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Art Unit: 3724

Page 5

The razor blade of Hahn et al. as modified above shows all the claimed structure except the amorphous coating of Hahn et al. is formed of diamond-like carbon material (DLC) rather than amorphous diamond material as claimed.

Decker et al. shows the use of amorphous diamond coating for the cutting edge of a razor blade to harden the same.

It would have been obvious to one skilled in the art to further modify Hahn et al. by substituting the amorphous diamond coating of Decker et al. for the amorphous diamond-like carbon coating of Hahn et al. The modification is obvious since it would only involve substituting one known type of amorphous coating for another for hardening the cutting edge of a razor blade.

4. Claim 12 is rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) and Lane et al. (U.S. Patent No. 3,911,579) as applied to claim 10 above, and further in view of Bache et al. (U.S. Patent No. 4,933,058).

The razor blade of Hahn et al. as modified above shows all the claimed structure except the interlayer (58) comprises niobium rather than a chromium containing material.

Bache et al. shows a razor blade comprising an interlayer of chromium (see column 5, lines 57-59) between a substrate of the blade and a diamond-like carbon hard coating of the razor blade.



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It would have been obvious to one skilled in the art to further modify Hahn et al. by substituting the chromium interlayer of Bache et al. for the niobium interlayer of Hahn et al. The modification is obvious since it would only involve substituting one known type of adherent layer for another for providing a desired adhesion between the blade substrate and the blade hard coating.

**Remarks**

Applicant's arguments with respect to claims 1-2 and 4-30 have been considered but are moot in view of the new ground(s) of rejection.

**Point of Contact**

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Hwei-Siu C. Payer whose telephone number is 703-308-1405. The examiner can normally be reached on Monday through Friday, 7:00 am to 4:00 pm.

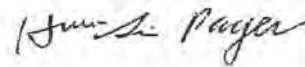
If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Allan N. Shoap can be reached on 703-308-1082. The fax phone numbers for the organization where this application or proceeding is assigned are 703-305-3579 for regular communications and 703-305-3579 for After Final communications.

Application/Control Number: 09/515,421  
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Page 7

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-1148.

H Payer  
February 4, 2002



**Hwei-Bin Payer**  
**Primary Examiner**





COPY OF PAPERS  
ORIGINALLY FILED

Attorney's Docket No.: 00216-483001 / Case 8073

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Title : RAZOR BLADE TECHNOLOGY

Art Unit : 3724  
Examiner : Hwei-Siu Payer

Commissioner for Patents  
Washington, D.C. 20231

RECEIVED  
JUN 21 2002  
TECHNOLOGY CENTER MAIL  
#13/C  
6/28/02  
Hant

RESPONSE

In response to the action mailed February 5, 2002, please amend the application as follows:

In the claims:

Please amend claims 10, 20, 21, 29 and 30 as follows:

C1

-- 10. (Amended) The blade of claim 1 further comprising an interlayer between said substrate and said layer of hard coating.

C2

20. (Amended) The blade of claim 1, 8, 16 or 17 wherein said cutting edge has less rounding with repeated shaves than it would have without said overcoat layer.

C3

21. (Twice Amended) A shaving razor comprising  
a handle,  
a housing connected to said handle, and  
at least one razor blade mounted in said housing, said blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent  
facets,

CERTIFICATE OF MAILING BY FIRST CLASS MAIL

I hereby certify under 37 CFR §1.8(a) that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage on the date indicated below and is addressed to the Commissioner for Patents, Washington, D.C. 20231.

Date of Deposit June 6, 2002

Signature

Typed or Printed Name of Person Signing Certificate  
Jennifer Leveille

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 3

Attorney's Docket No.: 00216-483001 / Case 8073

#### REMARKS

Claims 10, 20, 21, 29 and 30 have been amended as indicated in the Office Action and are submitted to comply with 35 USC 112, second paragraph.

The invention, as claimed in independent claims 1, 21 and 25, is directed to a razor blade having a substrate with a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of chromium containing material thereon, and an outer layer of polytetrafluoroethylene (PTFE) thereon. The claims recite that the hard coating is made of "amorphous material," and, so limited, exclude crystalline material. Such amorphous material includes "diamond-like carbon" ("DLC"), as recited in dependent claim 4, and "amorphous diamond material," as recited in dependent claim 5. As noted at page 4, lines 2-4, with reference to the "Handbook of Physical Vapor Deposition (PVD) Processing," "DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond."

In the claimed invention, the use of an amorphous hard coating is advantageous in obtaining the desired tip geometry. When depositing a crystalline hard coating, the crystals can grow to a size that is larger than the desired tip radius, frustrating efforts to obtain a desired coating thickness and tip radius. This problem does not exist when depositing amorphous material, making it easier to deposit the desired amorphous hard coating thickness and obtain the desired tip radius shape. Such amorphous materials have been used extensively with a telomer (PTFE) outer coating, as described in the cited Hahn patent. A problem that has been noted with at least some amorphous carbon hard coating materials used with PTFE (in particular amorphous DLC), however, is that there is substantial rounding of the blade tips with repeated use during wet shaving, resulting in an increase in required cutting force during shaving.

The inventors found that, by using a chromium containing layer between an amorphous hard coating material and a PTFE layer, they could maintain the good edge strength provided by hard coating and have reduced tip rounding with repeated shaves. The razor blade has excellent shaving characteristics from the first shave onwards. These advantages are described in the application at page 2, line 27 to page 3, line 2. This is an unexpected result for the amorphous



Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
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Attorney's Docket No.: 00216-483001 / Case 8073

hard coating material, because it would not be expected that a material that is softer than the hard coating, namely the chromium containing layer, would provide wear resistance and/or additional strength to the hard coating.

Independent claims 1, 21, and 25 stand rejected as obvious under 35 USC 103(a) on the basis of Hahn U.S. Patent No. 5,295,305 in view of Lane U.S. Patent No. 3,911,579.

Hahn discloses depositing a PTFE layer directly on an amorphous hard coat carbon layer. This teaches the opposite of using a layer between the PTFE and amorphous hard coat layer, and Hahn thus teaches away from the invention in teaching that a PTFE layer should be deposited directly on the amorphous hard coat carbon layer.

Lane describes a blade having three layers deposited on a substrate, namely, refractory layer I, an adhesion promoting layer II containing chromium, and a PTFE layer III. The examples of refractory materials listed at col. 5, lines 56-64 are synthetic sapphire, corundum, glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride. The Kirk Othmer, Concise Encyclopedia of Chemical Technology (John Wiley & Sons 1999), p. 1732 (copy enclosed) states that "refractory materials" are generally understood to withstand temperatures above 1100°C, a characteristic that is met by the examples of refractory materials listed in Lane. Lane repeatedly stresses the use and advantages of refractory materials. Refractory materials are mentioned in the abstract, the first sentence of the Background (col. 1, lines 14, throughout the Background (e.g., at col. 1 lines 22-59 and col. 2, lines 41-42), throughout the "objects of the invention" at col. 2, lines 57-col. 3, line 8, throughout the Summary of Invention and preferred embodiments, and in all of the claims.

Amorphous materials, on the other hand, will degrade at much lower temperatures than the refractory materials to which Lane is directed; e.g., the diamond like carbon and amorphous diamond amorphous materials noted above would deteriorate at temperatures above 400°C, which is substantially less than the 1100°C lower limit for refractory materials.

Lane effectively teaches away from the use of a hard coating of an amorphous material as in the claimed invention by stressing instead that refractory materials should be used. As noted above, the use of an amorphous material is advantageous in the invention in terms of obtaining a desired tip geometry without the problems associated with crystalline hard coatings.

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
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There moreover is no motivation to combine, as Hahn teaches directly bonding the PTFE to the hard coating, thus teaching away from any use of an intermediate layer as in Lane. Lane emphasizes the use of refractory material, thus teaching away from any use of a non-refractory material as in Hahn.

Moreover, none of the references teach the unexpected result of providing reduced tip rounding with repeated shaves, i.e., increased strength and wear resistance, by providing a softer, chromium containing layer outside of the amorphous hard coating layer. Accordingly, the subject matters of claims 1, 21, and 25 are nowhere suggested by the combination of cited references, and the independent claims are allowable under 35 USC 102 and 103(a).

Attached is a marked-up version of the changes being made by the current amendment.

Applicant asks that all claims be allowed. Enclosed is a \$400 check for the Petition for Extension of Time fee. Please apply any other charges or credits to Deposit Account No. 06-1050.

Respectfully submitted,

Date: \_\_\_\_\_

June 6, 2002

William E. Booth

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Reg. No. 28,933

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20390340.doc



<b>Office Action Summary</b>	Application No. 09/515,421	Applicant(s) CLIPSTONE ET AL.	
	Examiner Hwei-Siu C. Payer	Art Unit 3724	

**-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --**

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

1) ☒ Responsive to communication(s) filed on 06 June 2002.

2a) ☒ This action is **FINAL**.                      2b) ☐ This action is non-final.

3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

4) ☒ Claim(s) 1, 2 and 4-30 is/are pending in the application.

4a) Of the above claim(s) \_\_\_\_\_ is/are withdrawn from consideration.

5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.

6) ☒ Claim(s) 1, 2 and 4-30 is/are rejected.

7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.

8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

9) ☐ The specification is objected to by the Examiner.

10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

11) ☐ The proposed drawing correction filed on \_\_\_\_\_ is: a) ☐ approved b) ☐ disapproved by the Examiner.

If approved, corrected drawings are required in reply to this Office action.

12) ☐ The oath or declaration is objected to by the Examiner.

**Priority under 35 U.S.C. §§ 119 and 120**

13) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

a) ☐ All   b) ☐ Some \* c) ☐ None of:

1. ☐ Certified copies of the priority documents have been received.

2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.

3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

14) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. § 119(e) (to a provisional application).

a) ☐ The translation of the foreign language provisional application has been received.

15) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121.

**Attachment(s)**

1) <input type="checkbox"/> Notice of References Cited (PTO-892)	4) <input type="checkbox"/> Interview Summary (PTO-413) Paper No(s). _____
2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)	5) <input type="checkbox"/> Notice of Informal Patent Application (PTO-152)
3) <input type="checkbox"/> Information Disclosure Statement(s) (PTO-1449) Paper No(s) _____	6) <input type="checkbox"/> Other: _____

Application/Control Number: 09/515,421  
Art Unit: 3724

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## Detailed Action

The amendment filed on June 6, 2002 has been entered.

### Claims Rejection - 35 U.S.C. 112, second paragraph

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

2. Claims 20, 29 and 30 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

(1) In claim 20, it is unclear how the cutting edge has less rounding with repeated shaves than it would have without the overcoat layer.

(2) In claims 29 and 30, "an equivalent RF bias scheme" is vague and indefinite.

### Claims Rejection - 35 U.S.C. 103(a)

1. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.



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2. Claims 1, 2, 4, 6-11 and 13-30 are rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) in view of Lane et al. (U.S. Patent No. 3,911,579).

Hahn et al. discloses a razor blade (Fig.3) comprising a substrate (50) with a cutting edge defined by a sharpened tip (70) and adjacent facets (62,64); a layer of hard coating (60) on said cutting edge, the hard coating (60) being made of amorphous material such as diamond-like carbon material (see column 3, lines 65-66); a niobium interlayer (58) between the substrate (50) and the hard coating (60); and a polytetrafluoroethylene outer layer (72) substantially as claimed except Hahn et al. lacks an overcoat layer of chromium between the hard amorphous coating (60) and the polytetrafluoroethylene outer layer (72).

Lane et al. shows a razor blade comprising an overcoat layer (II) of a chromium containing material provided between a layer of hard coating (I) and an outer layer of polytetrafluoroethylene (III) by RF sputtering process or DC sputtering process (see column 6, lines 26-32). The chromium overcoat layer (II) not only adheres strongly to the lubricious coating (III) but also provides a hard and durable shaving edge (see column 6, lines 11-22).

It would have been obvious to one skilled in the art to modify Hahn et al. by providing a layer of chromium coating between the hard coating (60) and the polytetrafluoroethylene layer to aid in facilitating a hard and durable shaving edge for the razor blade as taught by Lane et al.

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It is noted claims 14 and 16 call for the polytetrafluoroethylene outer layer (72) being Krytox LW 1200. To select a commercially available polytetrafluoroethylene such as Krytox LW 1200 from DuPont is deemed to be an obvious matter of preference and not patentably distinct.

Claims 13 and 28-30 call for the sputter depositing condition (i.e. applying a DC bias of more negative than -200 volts) of the chromium overcoat layer. The claimed depositing condition is not patentable distinct over Hahn et al. as modified, since the exact sputtering condition depends more upon the final desired shape of the deposited coating on the razor blade than on any inventive concept.

3. Claim 5 is rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) and Lane et al. (U.S. Patent No. 3,911,579) as applied to claim 2 above, and further in view of Decker et al. (U.S. Patent No. 5,799,549).

The razor blade of Hahn et al. as modified above shows all the claimed structure except the amorphous coating of Hahn et al. is formed of diamond-like carbon material (DLC) rather than amorphous diamond material as claimed.

Decker et al. shows the use of amorphous diamond coating for the cutting edge of a razor blade to harden the same.

It would have been obvious to one skilled in the art to further modify Hahn et al. by substituting the amorphous diamond coating of Decker et al. for the amorphous diamond-like carbon coating of Hahn et al. The modification is obvious since it would



Application/Control Number: 09/515,421  
Art Unit: 3724

Page 5

only involve substituting one known type of amorphous coating for another for hardening the cutting edge of a razor blade.

4. Claim 12 is rejected under 35 U.S.C. 103(a) as being unpatentable over Hahn et al. (U.S. Patent No. 5,295,305) and Lane et al. (U.S. Patent No. 3,911,579) as applied to claim 10 above, and further in view of Bache et al. (U.S. Patent No. 4,933,058).

The razor blade of Hahn et al. as modified above shows all the claimed structure except the interlayer (58) comprises niobium rather than a chromium containing material.

Bache et al. shows a razor blade comprising an interlayer of chromium (see column 5, lines 57-59) between a substrate of the blade and a diamond-like carbon hard coating of the blade.

It would have been obvious to one skilled in the art to further modify Hahn et al. by substituting the chromium interlayer of Bache et al. for the niobium interlayer of Hahn et al. The modification is obvious since it would only involve substituting one known type of adherent layer for another for providing a desired adhesion between the blade substrate and the blade hard coating.

#### **Remarks**

Applicants argues, at page 4 of the amendment, the refractory layer of Lane (et al.) is of refractory material which is not "a hard coating of an amorphous material" as claimed, since refractory materials are to withstand temperature above 110° C while

Application/Control Number: 09/515,421  
Art Unit: 3724

Page 6

amorphous material will degrade at much lower temperatures than the refractory materials.

Applicants' attention is directed to pages 62-63 of column 5 in the Lane et al. '579 reference which states "glass" (i.e. amorphous material) can be used. Further, Lane et al. further states that it must be recognized this invention is not necessarily limited to refractory materials (see column 5, lines 66-68).

**Action Made Final**

**THIS ACTION IS MADE FINAL.** Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire **THREE MONTHS** from the mailing date of this action. In the event a first reply is filed within **TWO MONTHS** of the mailing date of this final action and the advisory action is not mailed until after the end of the **THREE-MONTH** shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than **SIX MONTHS** from the mailing date of this final action.



Application/Control Number: 09/515,421  
Art Unit: 3724

Page 7

**Point of Contact**

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Hwei-Siu C. Payer whose telephone number is 703-308-1405. The examiner can normally be reached on Monday through Friday, 7:00 am to 4:00 pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Allan N. Shoap can be reached on 703-308-1082. The fax phone numbers for the organization where this application or proceeding is assigned are 703-305-3579 for regular communications and 703-305-3579 for After Final communications.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-1148.

H Payer  
August 8, 2002

*H Payer*

By: Hwei-Siu C. Payer  
Date: August 8, 2002



Attorney's Docket No.: 00216-483001 / Case 8073

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Title : RAZOR BLADE TECHNOLOGY

Art Unit : 3724  
Examiner : Hwei-Siu Payer

BOX AF  
Commissioner for Patents  
Washington, D.C. 20231

RECEIVED

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TECHNOLOGY CENTER R3700

RESPONSE

In response to the action mailed August 8, 2002, please amend the application as follows:

In the claims:

Please cancel claims 2, 22, 29 and 30.

Please amend claims 1, 4, 5, 21, and 23-25 as follows:

--1. (Amended) A razor blade comprising a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.

4. (Amended) The blade of claim 1 wherein said hard carbon coating comprises diamond-like carbon material.

CERTIFICATE OF MAILING BY FIRST CLASS MAIL

I hereby certify under 37 CFR §1.8(a) that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage on the date indicated below and is addressed to the Commissioner for Patents, Washington, D.C. 20231.

January 8, 2003  
Date of Deposit  
Signature Jennifer Leveille

Jennifer Leveille  
Typed or Printed Name of Person Signing Certificate



Applicant : Neville Sommerberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 2

Attorney's Docket No.: 00216-483001 / Case 8073

5. (Amended) The blade of claim 1 wherein said hard carbon coating comprises amorphous diamond material.

21. (Three Times Amended) A shaving razor comprising  
a handle,  
a housing connected to said handle, and  
at least one razor blade mounted in said housing, said blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.

23. (Amended) The razor of claim 21 further comprising a niobium interlayer between said substrate and said hard coating.

24. (Amended) The razor of claim 21 wherein said overcoat layer consists of chromium.

25. (Three Times Amended) A method of making a razor blade comprising  
providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
adding a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,  
adding an overcoat layer of a chromium containing material on said layer of hard coating,  
and  
adding an outer layer of polytetrafluoroethylene coating over said overcoat layer.--

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 3

Attorney's Docket No.: 00216-483001 / Case 8073

#### REMARKS

Claims 29 and 30 have been cancelled. It is submitted that claim 20 describes a physical property of the cutting edge and is definite.

Independent claims 1, 21 and 25 have been amended to recite an amorphous material containing carbon.

Independent claims 1, 21, and 25 stand rejected as obvious under 35 USC 103(a) on the basis of Hahn U.S. Patent No. 5,295,305 in view of Lane U.S. Patent No. 3,911,579.

The invention, as claimed in independent claims 1, 21 and 25, is directed to a razor blade having a substrate with a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of chromium containing material thereon, and an outer layer of polytetrafluoroethylene (PTFE) thereon. The claims recite that the hard coating is made of "amorphous material containing carbon" and, so limited, exclude crystalline material. Such amorphous material including carbon includes "diamond-like carbon" ("DLC"), as recited in dependent claim 4, and "amorphous diamond material," as recited in dependent claim 5. As noted at page 4, lines 2-4, with reference to the "Handbook of Physical Vapor Deposition (PVD) Processing," "DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond."

In the claimed invention, the use of an amorphous hard coating is advantageous in obtaining the desired tip geometry. When depositing a crystalline hard coating, the crystals can grow to a size that is larger than the desired tip radius, frustrating efforts to obtain a desired coating thickness and tip radius. This problem does not exist when depositing amorphous material, making it easier to deposit the desired amorphous hard coating thickness and obtain the desired tip radius shape. Such amorphous materials have been used extensively with a telomer (PTFE) outer coating, as described in the cited Hahn patent. A problem that has been noted with at least some amorphous carbon hard coating materials used with PTFE (in particular amorphous DLC), however, is that there is substantial rounding of the blade tips with repeated use during wet shaving, resulting in an increase in required cutting force during shaving.



Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
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Page : 4

Attorney's Docket No.: 00216-483001 / Case 8073

The inventors found that, by using a chromium containing layer between an amorphous hard coating material and a PTFE layer, they could maintain the good edge strength provided by hard coating and have reduced tip rounding with repeated shaves. The razor blade has excellent shaving characteristics from the first shave onwards. These advantages are described in the application at page 2, line 27 to page 3, line 2. This is an unexpected result for the amorphous hard coating material containing carbon, because it would not be expected that a material that is softer than the hard coating, namely the chromium containing layer, would provide wear resistance and/or additional strength to the hard coating.

Hahn discloses depositing a PTFE layer directly on an amorphous hard coat carbon layer. This teaches the opposite of using a layer between the PTFE and amorphous hard coat layer, and Hahn thus teaches away from the invention in teaching that a PTFE layer should be deposited directly on the amorphous hard coat carbon layer.

Lane describes a blade having three layers deposited on a substrate, namely, refractory layer I, an adhesion promoting layer II containing chromium, and a PTFE layer III. The examples of refractory materials listed at col. 5, lines 56-64 are synthetic sapphire, corundum, glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride. The Kirk Othmer, Concise Encyclopedia of Chemical Technology (John Wiley & Sons 1999), p. 1732 (copy enclosed) states that "refractory materials" are generally understood to withstand temperatures above 1100°C, a characteristic that is met by the examples of refractory materials listed in Lane. Lane repeatedly stresses the use and advantages of refractory materials. Refractory materials are mentioned in the abstract, the first sentence of the Background (col. 1, lines 14, throughout the Background (e.g., at col. 1 lines 22-59 and col. 2, lines 41-42), throughout the "objects of the invention" at col. 2, lines 57-col. 3, line 8, throughout the Summary of Invention and preferred embodiments, and in all of the claims.

Amorphous materials, on the other hand, will degrade at much lower temperatures than the refractory materials to which Lane is directed; e.g., the diamond like carbon and amorphous diamond amorphous materials noted above would deteriorate at temperatures above 400°C, which is substantially less than the 1100°C lower limit for refractory materials.

Lane effectively teaches away from the use of a hard coating of an amorphous material as in the claimed invention by stressing instead that refractory materials should be used. As noted



Applicant : Neville Sonnabend et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 5

Attorney's Docket No.: 00216-483001 / Case 8073

above, the use of an amorphous material is advantageous in the invention in terms of obtaining a desired tip geometry without the problems associated with crystalline hard coatings.

There moreover is no motivation to combine, as Hahn teaches directly bonding the PTFE to the hard coating, thus teaching away from any use of an intermediate layer as in Lane. Lane emphasizes the use of refractory material, thus teaching away from any use of a non-refractory material as in Hahn, and in particular teaches away from an amorphous material containing carbon, as claimed herein.

Moreover, none of the references teach the unexpected result of providing reduced tip rounding with repeated shaves, i.e., increased strength and wear resistance, by providing a softer, chromium containing layer outside of the amorphous hard coating layer.

In the office action, it is noted:

Applicants' attention is directed to pages 62-63, col. 5 in the Lane et al. '579 reference which states "glass" (i.e. amorphous material) can be used. Further, Lane et al. further states that it must be recognized this invention is not necessarily limited to refractory materials (see column 5, lines 66-68).

The cited passages from Lane follow:

It must be noted, however, that other generally classified refractory materials such as glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride amongst others may be successfully used in razor blade or cutting edge applications. It must be further recognized that this invention is not necessarily limited to refractory material but may find equal application to any material displaying desirable blade or cutting edge characteristics without having the necessary or preferred degree of adhesion to a subsequent lubricious coating. (Col. 5, line 61-col. 6, line 4)(emphasis added).

Lane calls glass a "generally classified refractory material" and thus is not teaching the use of amorphous materials other than glass. Moreover, all claims are explicitly limited to amorphous materials containing carbon, which are in no way suggested by Lane's teaching of refractory materials or by his mention of glass. Lane's further mention that the invention "may find equal application to any material" appears speculative, in light of everything else said in the document about the importance of refractory materials, and, in any event, is so general as to not point to any particular material, let alone amorphous materials containing carbon.



Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Page : 6

Attorney's Docket No.: 00216-483001 / Case 8073

Accordingly, the subject matters of claims 1, 21, and 25 are nowhere suggested by the combination of cited references, and the independent claims are allowable under 35 USC 103(a). The remaining claims depend on the independent claims and are allowable with them.

Attached is a marked-up version of the changes being made by the current amendment.

Applicant asks that all claims be allowed. Enclosed is a \$400 check for the Petition for Extension of Time fee. Please apply any other charges or credits to Deposit Account No. 06-1050.

Respectfully submitted,

Date: Jan 2, 2003

William E. Booth  
William E. Booth  
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20494428.doc



Attorney's Docket No.: 00216-483001 / Case 8073

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Neville Sonnenberg et al.  
Serial No. : 09/515,421  
Filed : February 29, 2000  
Title : RAZOR BLADE TECHNOLOGY

Art Unit : 3724  
Examiner : Hwei-Siu Payer

Mail Stop RCE  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

AMENDMENT

Prior to examination following the filing of a request for continued examination, please amend the application as indicated on the following pages.

CERTIFICATE OF MAILING BY FIRST CLASS MAIL

I hereby certify under 37 CFR §1.8(a) that this correspondence is being deposited with the United States Postal Service as first class mail with sufficient postage on the date indicated below and is addressed to the Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

August 6, 2003

Date of Deposit

Signature

Jennifer Leveille

Typed or Printed Name of Person Signing Certificate

GILLETTE-DSC-0220837



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Attorney's ~~Letter~~ No.: 00216-483001 / Case 8073

Amendments to the Claims:

This listing of claims replaces all prior versions and listings of claims in the application:

Listing of Claims:

1. (Currently amended) A razor blade comprising a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous material containing carbon,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.

21 [ Claims 2-3 (Canceled)

2 4. (Currently amended) The blade of claim [[2]] 1 wherein said hard carbon coating comprises diamond-like carbon material.

6 5. (Currently amended) The blade of claim [[2]] 1 wherein said hard carbon coating comprises amorphous diamond material.

11 6. (Original) The blade of claim 1 wherein said overcoat layer consists of chromium.

8 7. (Original) The blade of claim 1 wherein said overcoat layer consists of a chromium containing alloy compatible with polytetrafluoroethylene.

13 8. (Original) The blade of claim 4 wherein said overcoat layer consists of chromium.

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9. (Original) The blade of claim 7 wherein said alloy is a chromium platinum alloy.

10. (Original) The blade of claim 1 further comprising an interlayer between said substrate and said layer of hard coating.

11. (Original) The blade of claim 10 wherein said interlayer comprises niobium.

12. (Original) The blade of claim 10 wherein said interlayer comprises a chromium containing material.

13. (Original) The blade of claim 6, 7, 8, or 9 wherein said overcoat layer is compressively stressed.

14. (Original) The blade of claim 1 wherein said polytetrafluoroethylene is Krytox LW1200.

15. (Original) The blade of claim 4 further comprising a niobium interlayer between said substrate and said hard coating.

16. (Original) The blade of claim 8 wherein said polytetrafluoroethylene is Krytox LW1200.

17. (Original) The blade of claim 1 wherein said hard coating layer has a thickness less than 2,000 angstroms.

18. (Original) The blade of claim 1 wherein said overcoat layer is between 100 and 500 angstroms thick.

19. (Original) The blade of claim 1 wherein said outer layer is between 100 and 5,000 angstroms thick.



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18 20. (Original) The blade of claim 1, 8, 16 or 17 wherein said cutting edge has less rounding with repeated shaves than it would have without said overcoat layer.

21. (Currently amended) A shaving razor comprising  
a handle,  
a housing connected to said handle, and  
at least one razor blade mounted in said housing, said blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
a layer of hard coating on said cutting edge, said hard coating being made of amorphous  
material containing carbon,  
an overcoat layer of a chromium containing material on said layer of hard coating, and  
an outer layer of polytetrafluoroethylene coating over said overcoat layer.

22. (Canceled)

23. (Currently amended) The razor of claim [[22]] 21 further comprising a niobium interlayer  
between said substrate and said hard coating.

24. (Currently amended) The razor of claim 21 [[or 22]] wherein said overcoat layer consists of  
chromium.

25. (Currently amended) A method of making a razor blade comprising  
providing a substrate with a cutting edge defined by a sharpened tip and adjacent facets,  
adding a layer of hard coating on said cutting edge, said hard coating being made of amorphous  
material containing carbon,  
adding an overcoat layer of a chromium containing material on said layer of hard coating,  
and

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adding an outer layer of polytetrafluoroethylene coating over said overcoat layer.

(b) 26. (Original) The method of claim 25 wherein said adding a layer of hard coating includes vapor depositing a carbon containing material.

(26) 27. (Original) The method of claim 25 wherein said adding a layer of chromium containing material includes vapor depositing said chromium containing material.

(27) 28. (Original) The method of claim 27 wherein said adding a layer of chromium containing material includes sputter depositing under conditions to result in compressively stressed material.

Claim 29-30 (Canceled)

F (28) 31. (New) A razor blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent facets;  
a layer of a hard carbon containing material, doped with another element, on the cutting edge;  
an overcoat layer of a chromium containing material on the layer of a hard carbon containing material; and  
an outer layer of polytetrafluoroethylene over the overcoat layer.

(29) 32. (New) The razor blade of claim 31, wherein the element is a metal.

(30) 33. (New) The razor blade of claim 32, wherein the metal is selected from the group consisting of tungsten and titanium.

(31) 34. (New) The razor blade of claim 32, wherein the metal is chromium.



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35. (New) The razor blade of claim 34, wherein the hard carbon containing material is diamond-like carbon.

36. (New) The razor blade of claim 31, wherein the hard carbon containing material is selected from the group consisting of diamond-like carbon and amorphous diamond.

37. (New) The razor blade of claim 31, wherein the layer of hard carbon material has a thickness less than 2,000 angstroms, the overcoat layer has a thickness between 100 and 500 angstroms, and the outer layer has a thickness between 100 and 5,000 angstroms.

38. (New) A shaving razor comprising  
a handle,  
a housing connected to the handle, and  
at least one razor blade within the housing, the razor blade comprising  
a substrate with a cutting edge defined by a sharpened tip and adjacent facets;  
a layer of a hard carbon containing material, doped with another element, on the cutting edge;  
an overcoat layer of a chromium containing material on the layer of <sup>the</sup> hard carbon containing material; and  
an outer layer of polytetrafluoroethylene over the overcoat layer.

39. (New) The shaving razor of claim 38, wherein the element is a metal.

40. (New) The shaving razor of claim 38, wherein the metal is chromium.

41. (New) The shaving razor of claim 38, wherein the hard carbon containing material is selected from the group consisting of diamond-like carbon and amorphous diamond.

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42. (New) The blade of claim 1 wherein said hard coating is doped with another element.

43. (New) The razor of claim 2 wherein said hard coating is doped with another element.

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#### REMARKS

Amendments to claims 1-30 and the remarks herein related thereto were made in a response after final rejection that was filed on January 8, 2003. The amendments, however, were not entered in an Advisory Action dated January 22, 2003.

In addition, applicants present new claims 31-43 herein. These claims recite that the hard carbon containing material is doped with another element, as is supported in the application on page 3.

Claims 29 and 30 have been cancelled. It is submitted that claim 20 describes a physical property of the cutting edge and is definite.

Independent claims 1, 21 and 25 have been amended to recite an amorphous material containing carbon.

Independent claims 1, 21, and 25 stand rejected as obvious under 35 USC 103(a) on the basis of Hahn U.S. Patent No. 5,295,305 in view of Lane U.S. Patent No. 3,911,579.

The invention, as claimed in independent claims 1, 21 and 25, is directed to a razor blade having a substrate with a sharpened tip and adjacent facets, a layer of hard coating on the cutting edge, an overcoat layer of chromium containing material thereon, and an outer layer of polytetrafluoroethylene (PTFE) thereon. The claims recite that the hard coating is made of "amorphous material containing carbon" and, so limited, exclude crystalline material. Such amorphous material including carbon includes "diamond-like carbon" ("DLC"), as recited in dependent claim 4, and "amorphous diamond material," as recited in dependent claim 5. As noted at page 4, lines 2-4, with reference to the "Handbook of Physical Vapor Deposition (PVD) Processing," "DLC is an amorphous carbon material that exhibits many of the desirable properties of diamond but does not have the crystalline structure of diamond."

In the claimed invention, the use of an amorphous hard coating is advantageous in obtaining the desired tip geometry. When depositing a crystalline hard coating, the crystals can grow to a size that is larger than the desired tip radius, frustrating efforts to obtain a desired coating thickness and tip radius. This problem does not exist when depositing amorphous material, making it easier to deposit the desired amorphous hard coating thickness and obtain the



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desired tip radius shape. Such amorphous materials have been used extensively with a telomer (PTFE) outer coating, as described in the cited Hahn patent. A problem that has been noted with at least some amorphous carbon hard coating materials used with PTFE (in particular amorphous DLC), however, is that there is substantial rounding of the blade tips with repeated use during wet shaving, resulting in an increase in required cutting force during shaving.

The inventors found that, by using a chromium containing layer between an amorphous hard coating material and a PTFE layer, they could maintain the good edge strength provided by hard coating and have reduced tip rounding with repeated shaves. The razor blade has excellent shaving characteristics from the first shave onwards. These advantages are described in the application at page 2, line 27 to page 3, line 2. This is an unexpected result for the amorphous hard coating material containing carbon, because it would not be expected that a material that is softer than the hard coating, namely the chromium containing layer, would provide wear resistance and/or additional strength to the hard coating.

Hahn discloses depositing a PTFE layer directly on an amorphous hard coat carbon layer. This teaches the opposite of using a layer between the PTFE and amorphous hard coat layer, and Hahn thus teaches away from the invention in teaching that a PTFE layer should be deposited directly on the amorphous hard coat carbon layer.

Lane describes a blade having three layers deposited on a substrate, namely, refractory layer I, an adhesion promoting layer II containing chromium, and a PTFE layer III. The examples of refractory materials listed at col. 5, lines 56-64 are synthetic sapphire, corundum, glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride. The Kirk Othmer, Concise Encyclopedia of Chemical Technology (John Wiley & Sons 1999), p. 1732 (copy enclosed) states that "refractory materials" are generally understood to withstand temperatures above 1100°C, a characteristic that is met by the examples of refractory materials listed in Lane. Lane repeatedly stresses the use and advantages of refractory materials. Refractory materials are mentioned in the abstract, the first sentence of the Background (col. 1, lines 14, throughout the Background (e.g., at col. 1 lines 22-59 and col. 2, lines 41-42),



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throughout the "objects of the invention" at col. 2, lines 57-col. 3, line 8, throughout the Summary of Invention and preferred embodiments, and in all of the claims.

Amorphous materials, on the other hand, will degrade at much lower temperatures than the refractory materials to which Lane is directed; e.g., the diamond like carbon and amorphous diamond amorphous materials noted above would deteriorate at temperatures above 400°C, which is substantially less than the 1100°C lower limit for refractory materials.

Lane effectively teaches away from the use of a hard coating of an amorphous material as in the claimed invention by stressing instead that refractory materials should be used. As noted above, the use of an amorphous material is advantageous in the invention in terms of obtaining a desired tip geometry without the problems associated with crystalline hard coatings.

There moreover is no motivation to combine, as Hahn teaches directly bonding the PTFE to the hard coating, thus teaching away from any use of an intermediate layer as in Lane. Lane emphasizes the use of refractory material, thus teaching away from any use of a non-refractory material as in Hahn, and in particular teaches away from an amorphous material containing carbon, as claimed herein.

Moreover, none of the references teach the unexpected result of providing reduced tip rounding with repeated shaves, i.e., increased strength and wear resistance, by providing a softer, chromium containing layer outside of the amorphous hard coating layer.

In the office action, it is noted:

Applicants' attention is directed to pages 62-63, col. 5 in the Lane et al. '579 reference which states "glass" (i.e. amorphous material) can be used. Further, Lane et al. further states that it must be recognized this invention is not necessarily limited to refractory materials (see column 5, lines 66-68).

The cited passages from Lane follow:

It must be noted, however, that other generally classified refractory materials such as glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride amongst others may be successfully used in razor blade or cutting edge applications. It must be further recognized that this invention is not necessarily limited to refractory material but may find equal application to any material displaying desirable blade or cutting edge characteristics without having the

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necessary or preferred degree of adhesion to a subsequent lubricious coating.  
(Col. 5, line 61-col. 6, line 4)(emphasis added).

Lane calls glass a "generally classified refractory material" and thus is not teaching the use of amorphous materials other than glass. Moreover, all claims are explicitly limited to amorphous materials containing carbon, which are in no way suggested by Lane's teaching of refractory materials or by his mention of glass. Lane's further mention that the invention "may find equal application to any material" appears speculative, in light of everything else said in the document about the importance of refractory materials, and, in any event, is so general as to not point to any particular material, let alone amorphous materials containing carbon.

Accordingly, the subject matters of claims 1, 21, and 25 are nowhere suggested by the combination of cited references, and the independent claims are allowable under 35 USC 103(a). Dependent claims 4-20, 23-24 and 26-28 depend on these independent claims and are allowable with them.

New claims 31-43 also distinguish the prior art. They in particular recite that the hard carbon coating is doped with another element.

Applicants submit that all claims are in condition for allowance, and a Notice of Allowance is respectfully solicited.

Enclosed is a \$750 check for the fee for RCE. Please apply any other charges or credits to deposit account 06-1050.

Respectfully submitted,

Date: August 6, 2003

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US005795648A

**United States Patent** [19]

Goel et al.

[11] **Patent Number:** 5,795,648[45] **Date of Patent:** Aug. 18, 1998[54] **METHOD FOR PRESERVING PRECISION EDGES USING DIAMOND-LIKE NANOCOMPOSITE FILM COATINGS**5,087,434 2/1992 Frenklach et al. .  
5,094,915 3/1992 Subramaniam .  
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5,101,288 3/1992 Ohta et al. .[75] **Inventors:** Arvind Goel, Buffalo; Donald J. Bray, East Amherst, both of N.Y.

(List continued on next page.)

[73] **Assignee:** Advanced Refractory Technologies, Inc., Buffalo, N.Y.**FOREIGN PATENT DOCUMENTS**

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**OTHER PUBLICATIONS**[21] **Appl. No.:** 538,731[22] **Filed:** Oct. 3, 1995[51] **Int. CL<sup>6</sup>** B05D 3/06; B26B 21/00[52] **U.S. Cl.** 428/336; 428/408; 428/446;  
428/697; 428/457; 427/249; 427/255.2;  
427/255.3; 427/574; 427/578; 30/346.53;  
30/346.54; 30/346.55[58] **Field of Search** 428/408, 446,  
428/336, 457, 697; 30/346.53, 346.54,  
346.55; 427/249, 255.3, 574, 578[56] **References Cited****U.S. PATENT DOCUMENTS**4,191,735 3/1980 Nelson et al. .  
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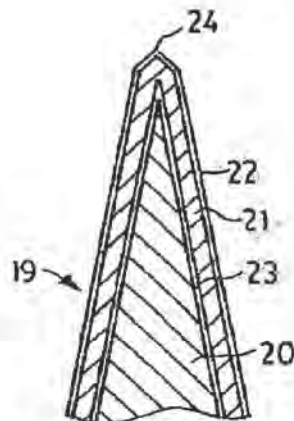
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*Primary Examiner*—Archene Turner*Attorney, Agent, or Firm*—Nixon, Hargrave, Devans & Doyle LLP[57] **ABSTRACT**

A method for preserving the precision-edges of a precision-edged substrate by applying to the substrate a corrosion resistant coating comprising a diamond-like solid state material having interpenetrating atomic scale networks comprising a first diamond-like carbon network stabilized by hydrogen, a silicon network stabilized by oxygen, and optionally at least one network made from dopant elements or dopant compounds containing elements from Groups 1-7b and 8 of the periodic table.

**28 Claims, 6 Drawing Sheets**

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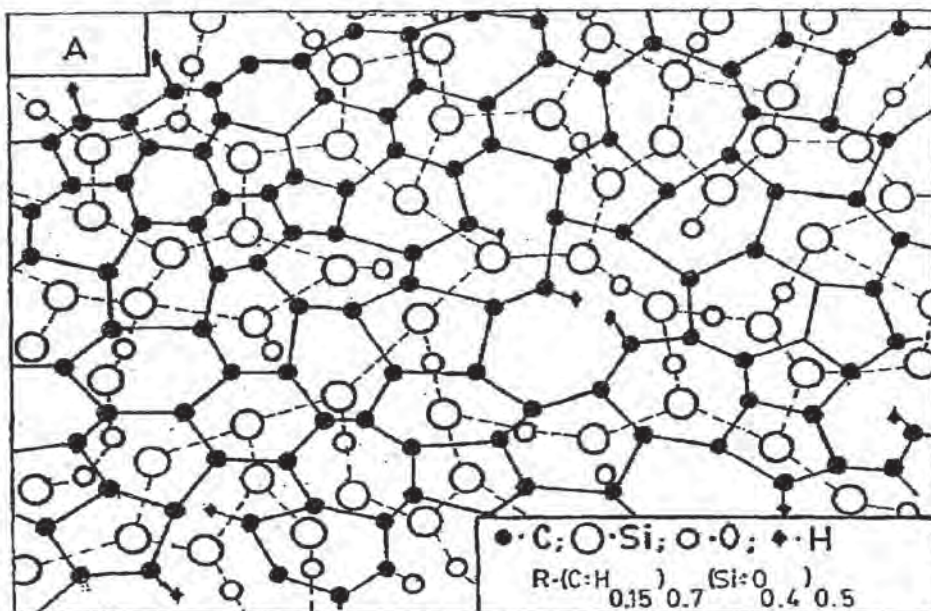


FIG. 1A

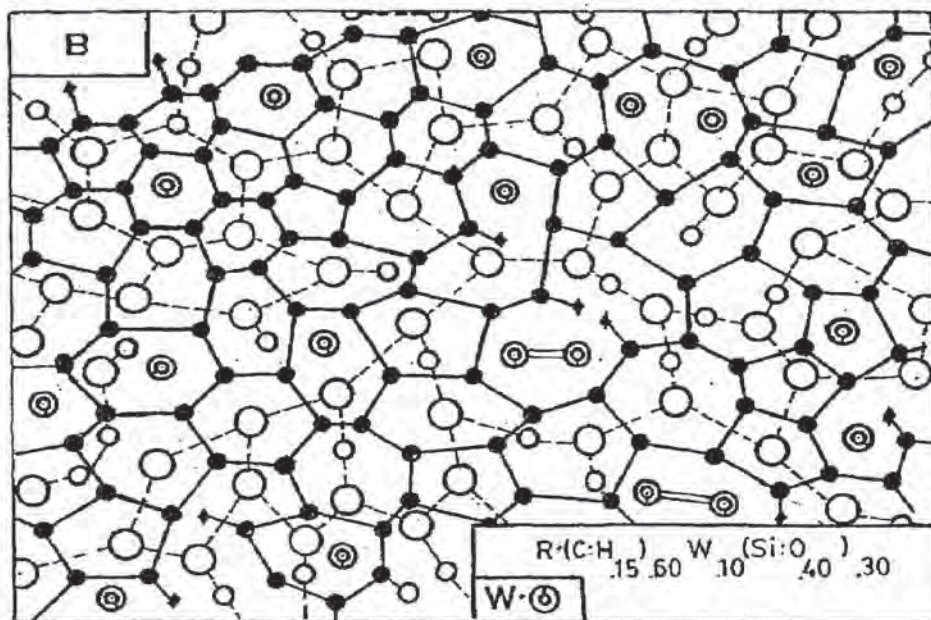


FIG. 1B

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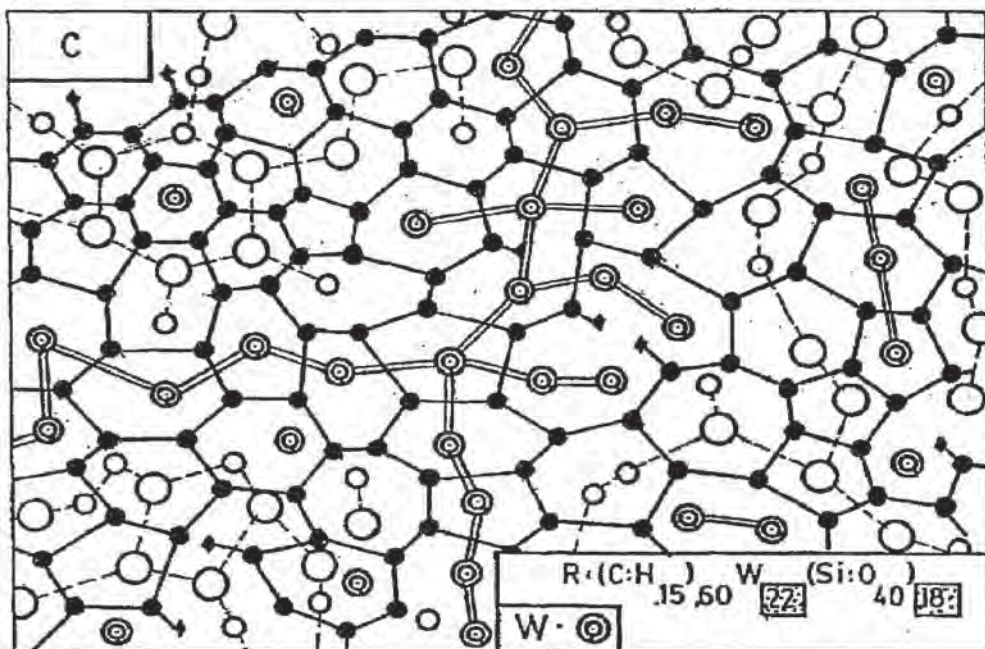


FIG. 1C



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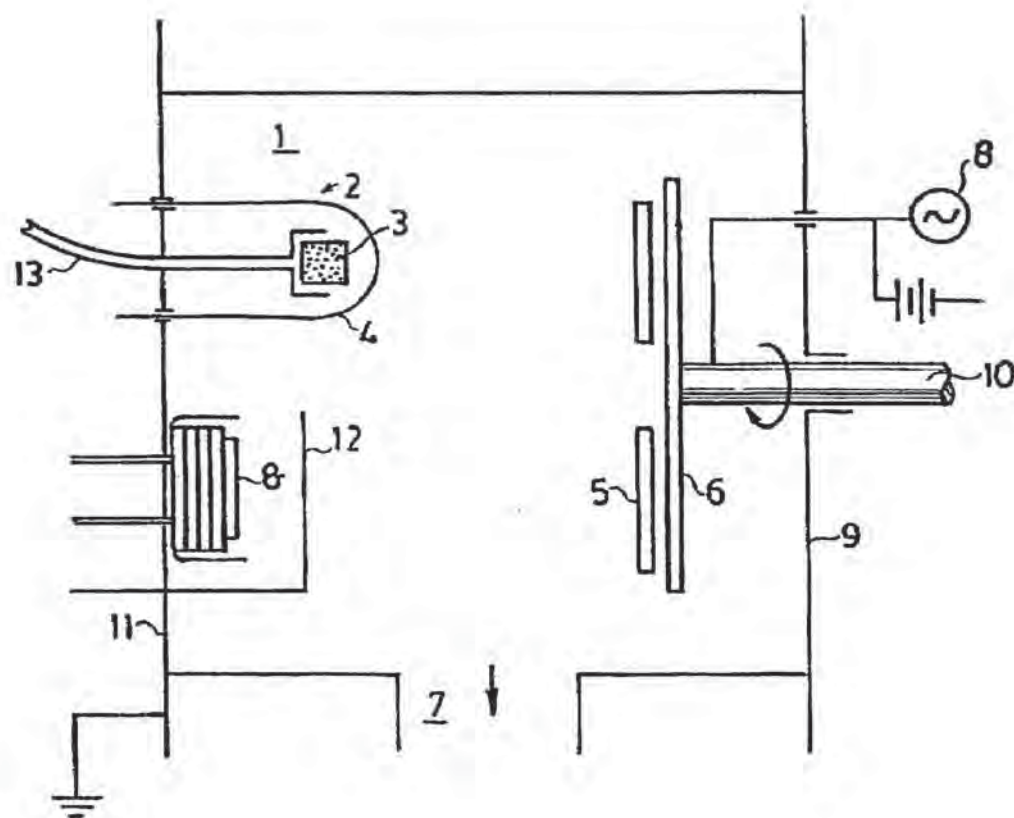


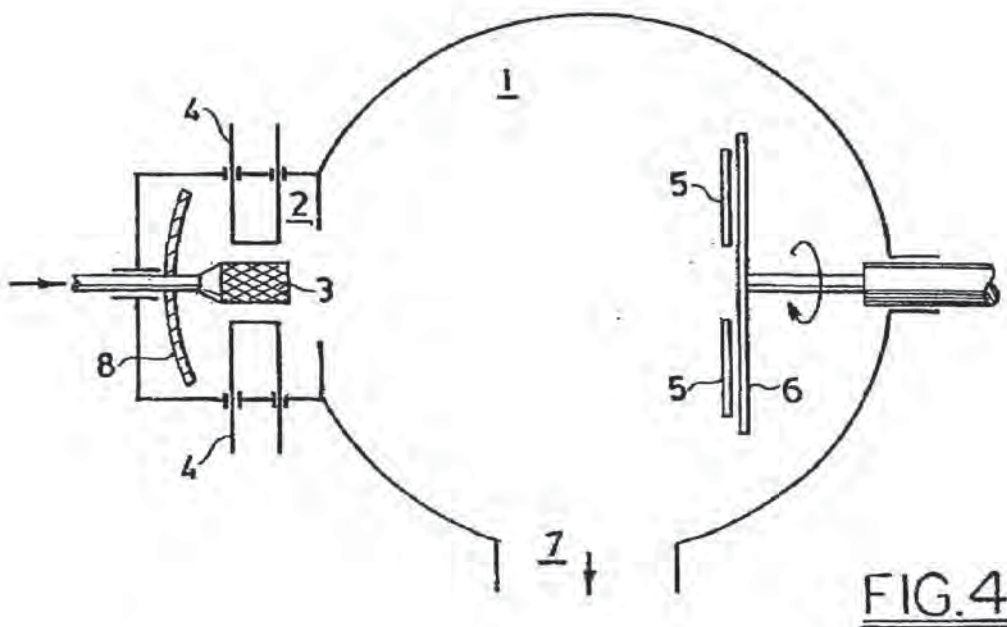
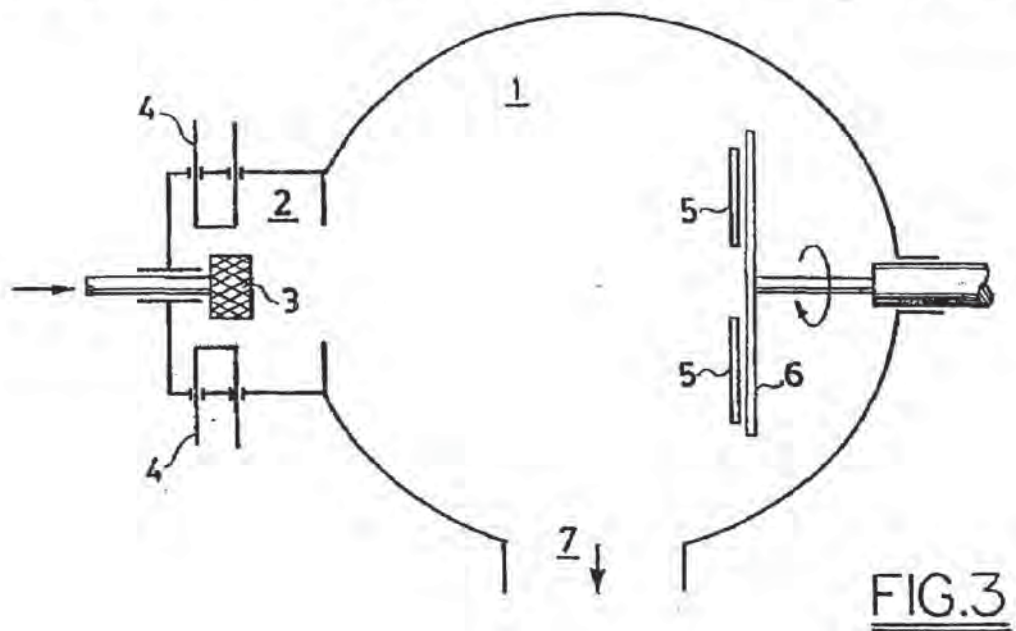
FIG. 2

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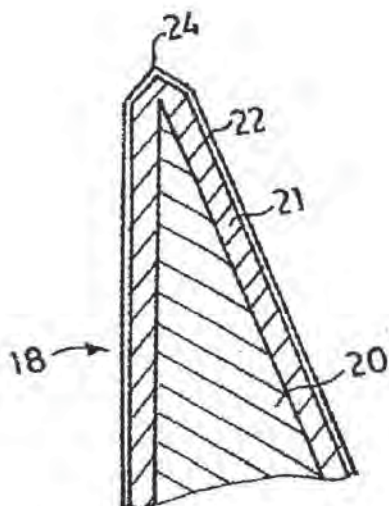


FIG. 5

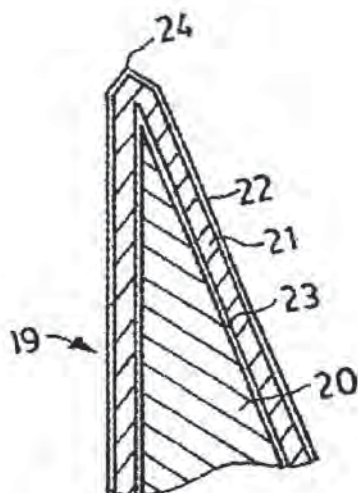


FIG. 6

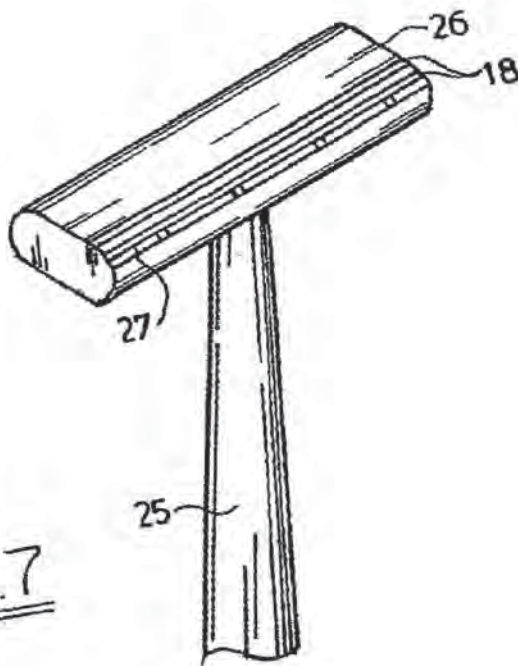


FIG. 7

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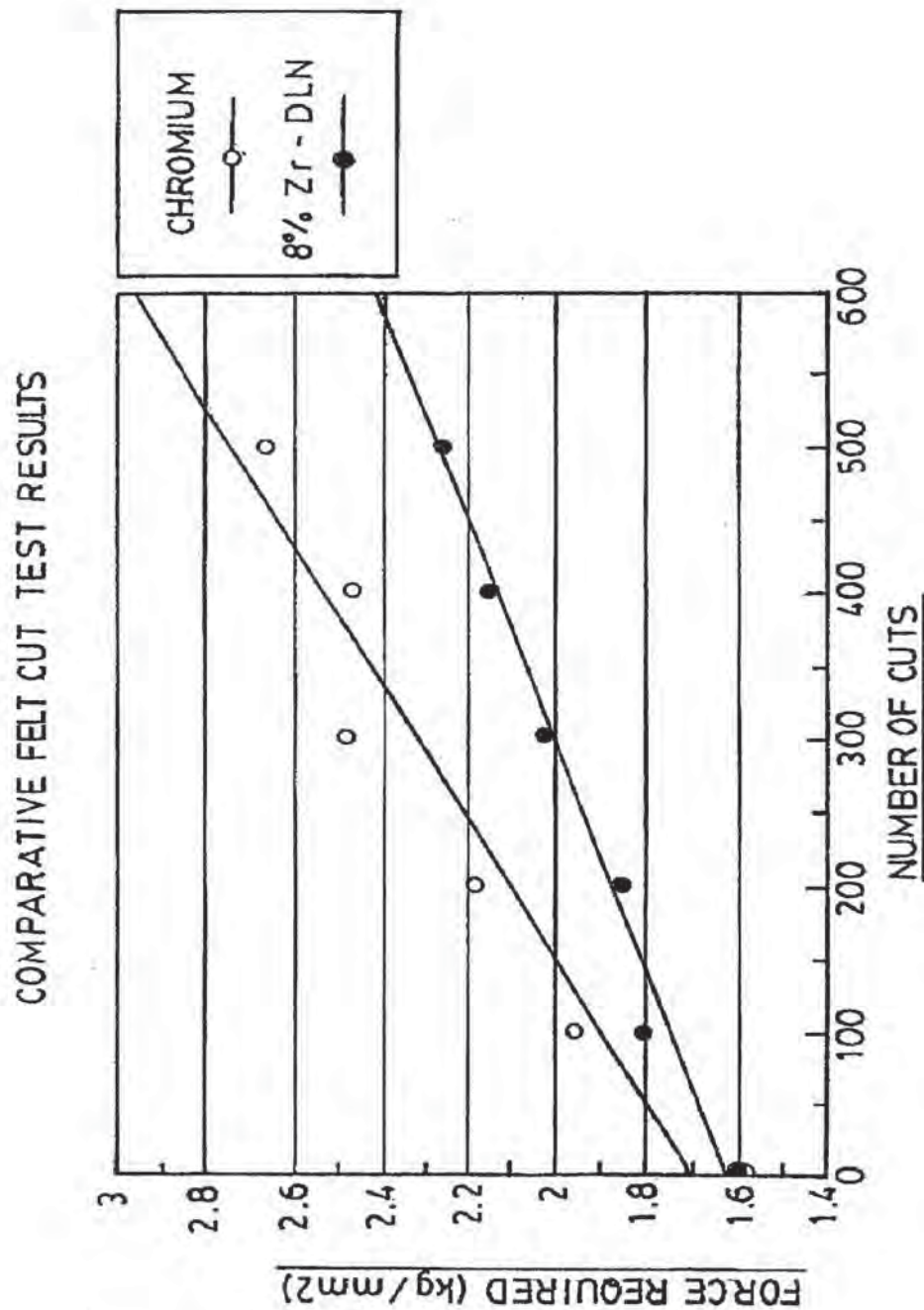


FIG. 8



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# METHOD FOR PRESERVING PRECISION EDGES USING DIAMOND-LIKE NANOCOMPOSITE FILM COATINGS

## FIELD OF THE INVENTION

The present invention relates to erosion resistant coatings, and especially precision-edge preserving coatings made from diamond-like materials used to keep substrate edges precise and sharp.

## BACKGROUND OF THE INVENTION

The preservation of sharp edges is important for many products and industries. Many bladed industrial and medical tools are only useful if they can have sharp edges which can be maintained for reasonably long periods of time. The sharpness which an edge has is the result of the precision of the edge formed by the substrate and any coatings thereon. Razor blades, for example, have an edge formed by producing a radius of curvature at the blade's extreme tip of from about 75 to about 1000 angstroms. For comparison purposes, a human hair has a width of about 100 micrometers. Such delicate precision substrate edges are often coated to preserve the precision of the edge for longer durations by attempting to inhibit the degradation of the edge.

Precision edge degradation can be caused by corrosive and/or erosive forces. Razor blades, for example dull quite easily; to an extent, immediately upon first use. Steel used for razor blades is therefore often coated first with a sputtered metal coating, followed by a coating of polytetrafluoroethylene (PTFE). While the PTFE coating is usually tens to thousands of angstroms thick, it appears to be substantially removed from the blade upon first use. Enough PTFE seems to survive to provide a measure of continued lubrication. However, the PTFE coatings do not appear to prevent the degradation of the precision edge.

Dulling of precision edges may be due to an increase in the radius of curvature at the blade's extreme tip, cracks, chips or breaks at the edge causing a jagged edge, erosion of edge material, or a combination of these factors. For razor blades, the degradation of the precision edge causes increased friction leading to user discomfort. Eventually the blade is replaced, or if a part of a disposable implement, the entire razor is simply discarded. For more expensive cutting implements in industrial or medical fields, etc., the dulling of precision-edged tools results in the need for sharpening or re-edging which takes time, requires the purchase of replacement equipment, and increases costs.

The depositing of harder material coatings has been tried in an attempt to preserve edge integrity. For many applications, the coating should also have excellent thermal stability; i.e. be able to withstand extreme heat, as from use itself (saw blade) or from sterilization procedures (autoclaving surgical tools).

Metal-based coatings such as steel, zinc, aluminum, chromium, nickel, alloys, cadmium, tantalum, palladium, boron, silicon, copper, gallium, rhenium, alloys thereof, etc. have demonstrated precision edge preservation and are used in many industries to provide protective coatings for sharp edges. However, coatings made from these materials are generally suitable only for metallic substrates.

Silicate based coatings are known to be resistant to air, acid, alkali, and gases at elevated temperatures. However, coatings made from silicates are not particularly strong materials and would not provide appropriate protection for precision edges.

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Certain ceramic materials used as coatings have displayed good corrosion resistance and could conceivably be used as edge preserving coatings. However, ceramics are brittle and subject to thermal shock failure. They are typically and rough and porous and would not provide the desired low friction.

Certain hard diamond-like coatings (DLCs), have been tried. However, a coating must not only be hard, but must have excellent adherence to the substrate being coated. Known DLCs often require an interlayer to adequately adhere them to a substrate. Ordinarily the presence of such an interlayer may not pose a problem. However, to preserve a precision edge, the total thickness of all deposited coatings must not appreciably increase the radius of curvature at the extreme tip of the edge which is very small. Further, the additional process of depositing interlayers between the DLC and the substrate increases the production cost. This can be significant, and even economically unsound for low cost items, such as disposable razors and disposable razor blades.

Therefore, a strong, hard, highly adherent, temperature, pH and chemical insensitive coating that can be applied to both metal and non-metal surfaces to preserve precision edges without applying interlayers, would be highly desirable.

## SUMMARY OF THE INVENTION

The present invention is directed to precision edge-preserving, corrosion and erosion resistant coatings made from a class of diamond-like materials, and substrates coated therewith. The diamond-like nanocomposite materials can be "tuned" or predictably and desirably altered by manipulating the chemical composition, to result in the best combination of properties, offering maximum edge preservation protection to the coated substrates.

In one embodiment, the present invention relates to a method for preserving precision edges of a precision-edged substrate, particularly a sharp-edged substrate, by providing a substrate to be coated and applying to the substrate a coating made from a class of diamond-like materials. The coatings are formed from interpenetrating networks comprising a first network of carbon in a diamond-like carbon network stabilized by hydrogen, a silicon network stabilized by oxygen and, optionally, at least one network of dopant elements, or dopant compounds containing elements from Groups 1-7b and 8 of the periodic table.

A still further embodiment of the present invention relates to a precision-edged apparatus comprising a precision-edged substrate coated with a precision edge-preserving coating. The coating applied to the substrate is made from a class of diamond-like material having interpenetrating atomic scale networks of carbon in a diamond-like carbon network stabilized by hydrogen, a glass-like silicon network stabilized by oxygen, and optionally at least one additional network of dopant elements or compounds containing elements selected from the group consisting of elements from Groups 1-7b and 8 of the periodic table.

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic diagram showing the principle microstructure of two-network (A), intermediate (B), and three-network (C) nanocomposites.

FIG. 2 is a schematic diagram detailing the main method of fabrication of the DLN coatings.

FIG. 3 is a schematic diagram detailing the methods of fabrication of DLN coatings using reflected beam flow.



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FIG. 4 is a schematic diagram detailing a preferred DLN fabrication and deposition chamber.

FIG. 5 is an enlarged cross-section view of a razor blade coated with the DLN coating.

FIG. 6 is an enlarged cross-section view of a razor blade coated with the DLN coating and an interlayer.

FIG. 7 is perspective view of a razor having DLN-coated razor blades incorporated into the head.

FIG. 8 is a graph of plotted values of the force required to make cuts in the wool felt cut test versus the number of cuts for chromium and DLN coated blades.

#### DETAILED DESCRIPTION OF THE INVENTION

The present invention is directed to precision edge-preserving, corrosion and erosion resistant coatings made from a class of diamond-like materials and substrates coated therewith. The diamond-like materials can be "tuned" or predictably and desirably altered by manipulating the amounts of substituent to result in the best combination of properties to offer maximum edge preservation protection to the precision-edged substrates.

In one embodiment, the present invention relates to a method for preserving precision edges of a substrate, particularly a sharp-edged substrate, by applying to the substrate a coating made from a class of diamond-like materials. The coatings are formed from interpenetrating networks comprising a first network of carbon in a diamond-like carbon network stabilized by hydrogen, a silicon network stabilized by oxygen and, optionally, at least one network of dopant elements, or dopant compounds containing elements from Groups 1-7b and 8 of the periodic table.

For the purposes of this application, a precision edge is understood to be the area of ultimate narrowing of a substrate, resulting in the convergence of two sides of the substrate to achieve a small radius of curvature at a tip. A small radius of curvature is understood to be one of from about 25 angstroms up to several microns. For very sharp blades, the radius of curvature is from about 75 angstroms to about 1000 angstroms. For other less sharp cutting tools, the radius at the tip may be up to hundreds of microns, while still being considered a precision edge.

Corrosion is defined as the electrochemical degradation of metals or alloys due to reaction with their environment, which is accelerated by the presence of acids or bases. In general, the corrodibility of a metal or alloy depends upon its position in the activity series. Corrosion products often take the form of metallic oxides or halides. In addition, corrosion may be considered to be the degradation of non-metal substrates by exposure to natural environmental conditions as well as exposure to organic materials.

In addition to the edge-preserving and corrosion-resistant properties of the coatings of the present invention, the coatings are strong and erosion resistant, such as to chemicals, abrasion, or ablation while also being highly thermally stable. The coatings would therefore be impervious to biological or chemical attack. The resistance of the coatings of the present invention to erosion, reduces the possibility of, for example, physical chipping. This results in the surface of the substrate being less likely to exposure to environmental corrosive forces. The coatings have excellent adherence to various substrates and are resistant to thermal shock at elevated temperatures beyond those known to erode known diamond-like coatings.

In one embodiment, FIG. 5 shows an enlarged cross-sectional view of a razor blade 18 with a precision-edged tip

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24 coated with the DLN coating of the present invention. In FIG. 5, a substrate 20 is coated with a layer of DLN 21. A thin coating of polytetrafluoroethylene (PTFE) 22 is shown deposited on the DLN coating 21.

FIG. 6 shows a coated blade 19 with a precision-edged tip 24 comprising a substrate 20 that has been coated with an interlayer 23. The interlayer 23 is then coated with the DLN coating 21, which is finally coated with the PTFE coating 22. The interlayer is a thin layer of material selected from silicon, silicon carbide, vanadium, tantalum, niobium, molybdenum and alloys thereof, alone or in combination with one another. The interlayers are deposited to a thickness of from about 50 to about 500 angstroms. The PTFE is deposited to a thickness of from about 10 angstroms to about 1000 angstroms, preferably from about 25 to about 75 angstroms.

In one embodiment, the blades may be assembled into a razor. FIG. 7 shows the blades 18 of FIG. 5 engaged in the head assembly 26 of a disposable razor 25. An opening 27 in the head allows debris to pass from the shaving plane. It is therefore understood that the DLN coated blades of the present invention may be manufactured as blades, such as replacement double-edged or single-edged blade, or may be incorporated into razor assemblies.

The fundamental structure of the preferred corrosion and erosion resistant atomic scale diamond-like nanocomposites (DLNs) used to coat the selected substrates is comprised of two or more self-stabilized random networks, each stabilized chemically by additional atomic species, while both networks also structurally stabilize each other. An example of a material with such a structure is the diamond-like nanocomposite (DLN) which is the subject of U.S. Pat. No. 5,352,493 and U.S. Ser. No. 08/249,167 filed May 24, 1994.

In the DLN, a random carbon network, mainly in the form of  $sp^3$  bonded carbon is chemically stabilized by hydrogen atoms, and a glass-like silicon network is chemically stabilized by oxygen atoms, resulting in a purely amorphous structure. "Amorphous" as used herein refers to a random structure or arrangement of atoms in a solid state that results in no long range regular ordering, and no crystallinity or granularity. The DLN materials have an amorphous structure and do not contain clusters greater than 10 Angstroms. This absence of clusters at the atomic scale is a characteristic of the DLN coatings of the present invention. Clusters can destroy amorphous nature of the structure, and can serve as active centers of degradation. Cluster formation is prevented in the sources, in the primary plasma, in the chamber space, and during film growth.

The atomic structure of the class of diamond-like nanocomposite (DLN) materials of the present invention is shown in FIG. 1(A). The materials may have one or more separate disordered networks of dopants, as shown in FIG. 1(B) and 1(C). The dopants may be any one or a combination of the transition metals and non-metals of the Groups 1-7b and 8 of the periodic table, and all three types of networks (C-H; Si-O and the dopant network, Me-Me) bonded to each other predominantly by weak chemical bonds. The network elements other than the C-H network may be referred to as alloying elements. Further, silicon and oxygen atoms may also be used in the dopant networks with other elements and compounds.

The DLN coatings of the present invention may comprise a two component network: the diamond-like carbon-hydrogen network interpenetrated with the glass-like silicon-oxygen network. A three component network may also be used comprising the Si-O and C-H networks with



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one or more dopant networks, with the dopants being interspersed with the previously mentioned two interpenetrating networks. In this instance three or more interpenetrating networks will be present in the DLN to form a so-called Me-DLN (metal-diamond-like nanocomposite) network. It is understood that non-metal dopant networks may be incorporated as the optionally present dopant networks interpenetrating the C-H and Si-O networks.

The three networks (C-H matrix, Si-O matrix and a dopant matrix) are bonded to one another mainly by weak chemical bonds. Carbide formation can be prevented even at metal concentrations as high as 50% (verified using Auger electron spectroscopy, electron spectroscopy for chemical analysis (ESCA), extended x-ray absorption fine structure spectroscopy (EXAFS) and Fourier transform infrared spectroscopy (FTIR)). Again, the properties of these materials can be varied over wide ranges depending on the dopant and the concentration selected, as well as the deposition technique and parameters. As already mentioned, the structure of these composites can be tailored at the molecular level. Therefore, unique electrical, optical, and other desirable solid state properties with desired mechanical strength, hardness and chemical resistance can be imparted on the DLN coatings.

Preferred dopant elements to be used in the Me-DLN network, and which are particularly effective for use as dopants in a corrosion-resistant Me-DLN coating are B, Si, Ge, Te, O, Mo, W, Ta, Nb, Pd, Ir, Pt, V, Fe, Co, Mg, Mn, Ni, Ti, Zr, Cr, Re, Hf, Cu, Al, N, Ag and Au; with W, Cr, Zr, Ti and Hf being preferred. Preferred compounds which may be used as dopants include TiN, BN, AlN, ZrN and CrN; with TiN and ZrN being most preferred.

The carbon content in the diamond-like nanocomposite is greater than about 40 atomic % of the DLN, preferably from about 40 to about 98 atomic %, more preferably from about 50 to about 98 atomic %. Although the DLN may theoretically be prepared without any hydrogen, the hydrogen content is preferably at least about 1 atomic % and up to about 40 atomic % of the carbon concentration. The sum of the silicon, oxygen and dopant elements and dopant containing compounds is greater than about 2 atomic % of the DLN. In one preferred embodiment, the ratio of carbon to silicon atoms is from about 2:1 to about 8:1, hydrogen to carbon atoms is about 0.01:1 to about 0.4:1, silicon to oxygen atoms is about 0.5:1 to about 3:1, and dopant to carbon atoms is about 0:1 to about 1.5:1. Therefore, in the DLN network, for every 1 part carbon, there is from about 0.01 to about 0.4 parts hydrogen, from about 0.125 to about 0.5 parts silicon, and from about 0.0375 to about 1.0 parts oxygen. In such a scheme, if a third dopant network were present, for every 1 part carbon, there would be from about 0.01 to about 1.5 parts dopants depending upon the desired characteristics to be imparted to the Me-DLN network.

The low intrinsic stress found in the DLNs contributes to their corrosion resistance properties. A coating must not only be unreactive to a corrosive agent, but should also act as a barrier layer, preventing contact between the corrosive agent and the protected substrate. DLC films typically possess high intrinsic stresses, and as a result usually suffer from pin holes and overall porosity. Due to the comparatively low stress present in DLN films and coatings, these coatings are pore-free, and therefore resist chemical attack and permeation.

The presence of the glass-like silicon network, stabilized by oxygen, serves to prevent the growth of graphitic carbon at high temperatures, to prevent metal cluster formation in

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metal-containing three-network nanocomposites, and reduce the internal stress in the nanocomposite structure and thereby enhance the adhesion to substrates. This appears to lead to superior adherence of the DLNs of the present invention to the substrate material.

As already mentioned, to improve adherence of coatings, DLC coatings often require an intermediate layer between the substrate and the DLC coating. Often, if the DLC coatings are too thick, delamination occurs. Surprisingly, with the DLN coatings of the present invention, adherence is so good that an interlayer is usually not required. As a result, the DLN coating may be applied directly to the substrate, and more thickly, without risking delamination from the substrate. The ability to apply a thicker layer of DLN coating results from the low intrinsic stress due to the Si-O network, and is believed to contribute to the superior erosion resistance of the DLN-coated substrates. Of course, interlayers may be used with the DLNs if desired. The tunability of the DLN structure also insures good adherence of the DLN to the interlayer as the DLN may be doped with a dopant to optimize compatibility and adherence to the interlayer as well as to the substrate. Such "tuning" is accomplished by incrementally altering the particular dopant as well as the dopant concentration. The DLNs may also have their properties altered when no dopants are included. In addition to altering chemical composition, changes in properties in the two-network DLN system also can be achieved by altering the deposition conditions in terms of temperature and pressure, etc. The DLNs therefore adhere well to both metal-containing and non-metal containing substrates.

The DLNs of the present invention have temperature stability far exceeding that of traditional diamond-like (DLC) materials. Crystalline diamond is stable to approximately 1100° C., upon which graphitization occurs. Quartz has long term thermal stability to 1470° C., and short term thermal stability up to 1700° C. Traditional, non-alloyed diamond-like (DLC) films are stable only to about 600° C. before graphitization occurs. By contrast, the DLN structures used to provide the corrosion and erosion resistant coatings of the present invention have long term stability to 1250° C. and short term stability to 2000° C. Therefore the thermal stability of the DLNs exceeds that of DLCs while preserving the amorphous, diamond-like state.

Further, in the range of from about 600° C. to about 1000° C., the chemical bonds of the carbon matrix of DLN materials partly change from  $sp^3$  to  $sp^2$ . However, the general structure of the nanocomposite and their "diamond-like" properties are preserved. By contrast, under similar conditions, the usual "diamond-like" carbon (DLC) is graphitized and loses its diamond-like properties. In the range of from 400° C. to 500° C. (preferably 430° C.), a reverse transition is observed, whereby the ratio of  $sp^3$  to  $sp^2$  is increased. It is believed that a varying percentage of the carbon in the DLNs is  $sp^3$  bonded carbon.

The density of the C-H and Si-O two network DLN varies from about 1.8 to about 2.1 g/cm<sup>3</sup>. The rest of the space is taken up by a random network of nanopores with diameters varying from about 0.28 to about 0.35 nm. The nanopore network does not form clusters or micropores. The properties of the two network DLN may then be tailored by adding dopant. The dopants fill the nanopore network in a random fashion, eventually resulting, at a certain dopant concentration, in an additional network without clusters or microcrystalline grains, even at concentrations as high as 50 atomic %. At concentrations below about 10 atomic %, the dopants are distributed as separate atoms in the nanopores of



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the diamond-like matrix. The average distance between dopant atoms in this quasi-random structure can be controlled by the concentration of the dopant. When the relative concentration of the dopant element or compound reaches about 20–25 atomic %, the dopants form the third (Me-Me) network in the DLN structure as shown in FIG. 1(C).

The electrical properties of the DLN structures of the present invention can be continuously varied over a wide magnitude (at least about 20 orders) from a highly dielectric state to a highly conductive state while preserving and improving the properties of the DLN state. A transition to a superconducting state, with the absence of electrical resistivity, is observed at low temperatures for certain three-network nanocomposite networks.

Another advantage of the DLNs of the present invention is their relative hardness and durability. The DLNs, especially the metal doped DLNs combine high microhardness with high elasticity. The microhardness values of the DLNs of the present invention range from about 5 to about 30 GPa.

The DLNs may be synthesized via co-deposition by clusterless beams of ions, atoms or radicals of the relevant elements, where the mean free path of each particle species exceeds the distance between its source and the growing particle film surface, and each beam contains particles of well-defined energy. Carbon-containing particle beams can be produced by plasma discharge in a plasmatron and extracted as charged particles by a high-voltage field in a vacuum chamber and directed onto the substrate.

FIG. 2 shows one preferred embodiment of the coating chamber used for the DLN coating deposition process. A vacuum deposition chamber 1 is provided to coat a substrate sample. A precursor inlet system 13, comprises a metal tube and a porous ceramic material 3 through which a liquid precursor, preferably a polysiloxane, is injected. The precursor inlet system 13 is shown incorporated into the chamber through the chamber base plate 11. The thermocathode 2 comprises a resistively heated thoriated tungsten filament 4. Substrates, 5 to be coated with DLN film are attached to the substrate holder 6. The power supply 8 is used for biasing the substrates (DC or RF). In practice the system is "pumped down" using normal vacuum pump down procedures. A gate valve (not shown) located on port 7 is closed and the system is backfilled with dry air, nitrogen or argon until the chamber reaches atmospheric pressure. The door of the chamber, 9, is then opened and substrate to be coated 5 are attached to the substrate holder 6 using any of many possible methods (spring clip, screw, clamp, etc.). Special fixtures may be required for substrates of special shapes. The substrate holder is designed in a way that it will also hold a cylinder sample (not shown), which, in operation, rotates both about the axis of the central drive shaft 10, and its own axis which is perpendicular to 10. In this way, the axis of the cylinder would be perpendicular to the axis of 10.

When the substrates, for example razor blades either singly or in a stacked arrangement, are loaded, the door of the chamber is closed, the chamber evacuated, and the gate valve opened to bring system pressure down to at least  $10^{-5}$  to  $10^{-6}$  Torr, which is the desired range of system base pressure. When the above base pressure is achieved, argon gas is introduced into the chamber via a needle valve or mass flow controller, until the chamber pressure reaches approximately  $5 \times 10^{-5}$  to  $1 \times 10^{-3}$  Torr, preferably about  $1-3 \times 10^{-4}$  Torr. The filament current, the filament bias and the electromagnet power supply are then switched on. The filament current is the current that passes through the thermocathode (also called the filament or the cathode). The filament bias is

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the constant floating voltage applied to the filament (approximately  $-150\text{V}$  in relation to ground). Plasma current is measured as the current between the filament and the base plate or ground. This voltage provides the field that moves electrons emitted by the filament to the base plate 11. The electromagnet power supply provides current to the electromagnet, which creates a magnetic field that results in the electron path becoming a spiral, increasing the electron path length and improving the probability of collisions between the electrons and the vapor molecules created due to precursor evaporation. The substrate bias power supply is concurrently switched on.

Switching on these power supplies results in creation of an argon plasma, which is used to clean the substrates prior to deposition. After the required duration of cleaning, the precursor supply is opened. Precursor flow is controlled via a needle valve and occurs due to the difference in pressure between the chamber and the outside atmosphere. When precursor flow and vaporization in the chamber has stabilized, the argon gas flow is turned off. The ionized precursor vapors form a plasma, ions from which are accelerated towards the substrate holder due to the substrate bias. Thus, deposition of DLN film onto the substrate occurs.

Co-deposition of a dopant material is carried out as follows. Argon flow to the magnetron is commenced and the magnetron 8 is switched on after the base pressure has been reached. A shutter 12 is used to prevent deposition while the substrate is cleaned via sputtering. When cleaning has been accomplished, the shutter is opened and sputtering is carried out at the desired power level. This may occur prior to commencement of DLN film deposition, during DLN film deposition, after DLN film deposition, or intermittently during DLN film deposition, depending on what kind of film structure and composition to be deposited are desired. Using DC or RF sputtering, materials of all kinds (metals, ceramics, alloys, etc.) can be used for co-deposition.

The growth conditions for nanocomposite films are as follows, with reference to FIG. 2. The pressure in the deposition chamber 1 should not exceed  $10^{-3}$  torr, with the pressure in the active zone of the plasma generation 2, in the range from about  $1.0 \times 10^{-3}$  to about  $5.0 \times 10^{-2}$  torr. The temperature of the substrate should not exceed about  $200^\circ\text{C}$ . with the temperature of the cathode filaments being in the range from about  $2100^\circ$  to about  $2950^\circ\text{C}$ . The current in the cathode filament is from about 70 to about 130A, with the voltage across the filament being from about 20 to about 30 V. The voltage with respect to the ground is from about 70 to about 130 V with the plasma current being from about 0.5 to about 20.0A. The voltage of the substrate holder is from about 0.1 to about 5.0 Kv, with all the carbon-containing and Si-containing species having kinetic energy in the range of from about 100 to about 1200 eV and from about 25 to about 300 eV respectively. The metal beams consist of free atoms or monatomic ions. The kinetic energy of the metal atoms/ions does not exceed from about 25 eV. With a precursor flow rate from about 0.5 to about 5.0 cc/hour, the growth rate of the DLN is from about 0.1 to about 2.0 micrometers/hour.

The preferred range of operation for most applications is a pressure of about  $1-3 \times 10^{-4}$  Torr, a plasma current of about 1 amp., a filament current of from about 60 to about 75 amp., a substrate voltage of from about 600 to about 1000 V DC, or forward power of about 100W in RF mode. The preferred frequency for RF mode is from about 90 to about 300 KHz. The preferred magnetron power depends on the type of material, composition and structure desired for the DLN coating.

In a further preferred embodiment, a plasma discharge in a triode plasmatron is used for DLN deposition, as shown



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schematically in FIG. 3, with the plasma energy density above about 5 Kwh/gram-atom of carbon. The charged particles are extracted by a high voltage field in the vacuum chamber and directed onto the substrate. It is preferable that the potential of the substrate holder is from about -0.3 to about +5.0 Kv, and most preferably 1.0+/-0.2 Kv for DC and RF. In the RF mode, the frequency is in the range of from about 0 to about 25 Mhz, and preferably from about 90 to about 300 kHz for RF. The ratio of the electron emission to the carbon precursor flow in the plasmatron is from about 0.5 to about 1.5 electrons per particle.

Organosilicon compounds, such as siloxane, are preferred precursors for C, H, Si and O. One preferred organosilicon compound is polyphenylmethylsiloxane, containing 1 to 10 Si atoms. The high boiling point siloxanes may be introduced directly into the active plasma region through a porous ceramic or metallo-ceramic (3 in FIGS. 3 and 4) which is heated via radiation thermocathodes 4. The photon and electron emission of the thermocathodes affect the evaporation, fragmentation and ionization of the precursor molecules on the surface of the ceramic, which thereby functions as an ion source for the plasma generator. An alternative method for injection of the siloxane precursors is to use direct injection from a diffusion pump.

The formation of dopant-containing beams may be realized by any one of, or combination of, the following methods: 1) thermal evaporation; 2) ion-sputtering; 3) ion beams. The dopant-containing beams are directed onto the growing film surface through the vacuum chamber to exclude interparticle collisions in the deposition chamber itself. Substrates are placed in an adjacent chamber on a rotating substrate holder, (for example a drum) which ensures double rotary motion, said adjacent chamber being connected to the plasma generation chamber by an opening for the emission of the atomic or ionic beams, as shown schematically in FIG. 3. Alternatively, the plasma generation may be carried out within the chamber containing the substrates (FIG. 2). A DC or a radio frequency potential is generally applied to the substrates during the deposition process. No external substrate heating is required. The substrate holder may be designed specifically to hold parts of different shapes such as cylinders, as would be apparent to one skilled in the field. Useful variation of the above described methods for deposition of DLN films include the use of sputtered silicon and oxygen gas as precursors for the Si and O<sub>2</sub>, the use of sputtered carbon and hydrogen or hydrocarbon gas used as carbon and hydrogen precursors, or any combination thereof.

For deposition on non-conducting substrates, such as plastic, a method whereby a flow of neutral radicals is reflected from a high voltage target and directed to the substrate as shown schematically in FIG. 4. The process employs depositions similarly to those shown in FIG. 3, except that a reflecting electrode is used to generate a neutral beam. This process eliminates surface damage of the substrate resulting from charged and/or fast particles impinging on the substrate during growth.

Extremely uniform and nonporous thin dielectric films may be deposited according to the present invention. The thickness of the deposited DLN coating has no theoretical limit. Existing technology and available equipment have allowed atomic-scale composite films and coating thicknesses typically in the range from about tens of angstroms up to 10 micrometers. The thickness of DLN deposited to adequately protect a sharp edge will depend upon the nature of the substrate. Very small sharp blades may only require DLN coatings from about 5 nanometers to about 150

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nanometers, while other apparatuses may require a protective DLN layer which is several micrometers (microns) thick. Therefore, the above-described DLN coatings of the present invention may be deposited on the selected substrate, or on interlayers if desired, in thicknesses ranging from about 5 nanometers to about 12 micrometers, preferably from about 20 nanometers to about 12 micrometers, depending only on the desired application of the coated substrate.

The deposition may be tailored or "tuned" to meet the properties required for a particular application. The random interpenetrating of the two- or three-network DLNs guarantees uniform strength of the structures in all directions. The structures are free of micropores even at thicknesses of about 80 Angstroms (8 nm). The DLNs are therefore extremely stable and possess unique combinations of chemical, mechanical, electronic, and superconducting properties.

Many uses for the precision edge-preserving DLN coatings of the present invention exist, including but not limited to the coating of metals and non-metals, surgical instruments, razor blades, industrial and non-industrial tools, cutlery, knives, pocket knives, and any precision-edged substrates which are vulnerable to corrosive and/or erosive attack, and dulling. The following examples serve only to further illustrate aspects of the present invention and should not be construed as limiting the invention.

#### EXAMPLE 1

##### Deposition of Undoped DLN Coatings on Razor Blades

One set of 2000 razor blades was coated on both sides with undoped DLN. The razor blades were mounted on a steel fixture with the blade edges facing the deposition sources (the blades held parallel to the beam). Deposition was carried out at a pressure of  $1.1 \times 10^{-4}$  Torr, a plasma current of 1.0-1.1 amp, and an RF load power of 125W. The deposition took place for 30 minutes. Shutters were used to shield the substrates during startup and shutdown of the plasmatron. At the deposition rate of 0.7 micrometers/hr, the test run resulted in a deposited DLN thickness of 3000 angstroms (0.3 micrometers) on a blade surface held flat facing the deposition beam, and a 300 angstrom film on a surface held at a 10° angle to the deposition beam. The thickness on the ultimate blade tip was approximately 3000-5000 angstroms, which was too high, resulting a dulling of the blade.

#### EXAMPLE 2

A second coating run nearly identical to that described in Example 1 was conducted, except that the test time was 10 minutes. The shorter test time resulted in a deposited DLN coating thickness on the blade edges of approximately 300-500 angstroms. The radius of the blade tip after coating was 200-300 angstroms.

#### EXAMPLE 3

##### Deposition of Doped DLN Coatings

Additional blades were coated with Zr-DLN and W-DLN under the following chamber conditions. RF bias frequency was 100-250 kHz, the load power was 80-120W, forward power was 100-150W, with the tungsten (W) and zirconium (Zr) doping at from about 10-20%.

#### EXAMPLE 4

##### Wool Felt Cutting Test

The blades coated according to the procedures of Examples 1-3 were tested by applying the blades against



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wool felt and cutting the felt 500 times. The difference between the force required to make the cut the first and last (500th) time was determined. A lower cutting force was required by the blades coated with the DLN versus chromium coated blades having a PTFE layer of 2000–3000 angstroms. See FIG. 8.

#### EXAMPLE 5

##### Mechanical Properties of DLN Films

High hardness and mechanical modulus measurements were obtained on 9 different compositions of DLN and doped-DLN films. Measurements were carried out using a nanoindenter (Nanoindenters, Knoxville, Tenn.). Hardness ranged from about 6 to about 21 GPa. Elastic modulus of from about 60–220 GPa was achieved. Hardness/modulus degradation in the films was minimal after exposure to 500° C.

Many other modifications and variations of the present invention are possible to the skilled practitioner in the field in light of the teachings herein. It is therefore understood that, within the scope of the claims, the present invention can be practiced other than as herein specifically described.

What is claimed:

1. A method for preserving a precision edge of a precision-edged substrate comprising:

providing a precision-edged substrate, wherein the precision-edged substrate is a cutting apparatus; and applying to said substrate a coating made from a diamond-like solid state material formed from interpenetrating networks comprising a diamond-like carbon network stabilized by hydrogen, a silicon network stabilized by oxygen, and optionally at least one network made from dopant elements or dopant compounds containing elements from Groups 1–7b and 8 of the periodic table.

2. The method according to claim 1, wherein the carbon, hydrogen, silicon and oxygen are obtained from the decomposition of an organosiloxane having from about 1 to about 10 silicon atoms.

3. The method according to claim 2, wherein the organosiloxane is polyphenylmethylsiloxane.

4. The method according to claim 1, wherein the carbon content of the coating is from about 40 wt. % to about 98 wt. %.

5. The method according to claim 1, wherein the carbon content of the coating is from about 50 wt.% to about 98 wt.%.

6. The method according to claim 1, wherein the carbon to silicon weight ratio of the coating is from about 2:1 to about 8:1.

7. The method according to claim 1, wherein the silicon to oxygen weight ratio of the coating is from about 0.5:1 to about 3:1.

8. The method according to claim 1, wherein the coating is deposited on a metal substrate.

9. The method according to claim 1, wherein the coating is deposited on a non-metal substrate.

10. The method according to claim 1, wherein the dopant elements are selected from the group consisting of B, Si, Ge, Te, O, Mo, W, Ta, Nb, Pd, Ir, Pt, V, Fe, Co, Mg, Mn, Ni, Ti, Zr, Cr, Re, Hf, Cu, Al, N, Ag, and Au.

11. The method according to claim 1, wherein the coating is deposited on the substrate to a thickness of from about 5 nm to about 12 micrometers.

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12. The method according to claim 1, wherein the coating is deposited on the substrate to a thickness of from about 5 nm to about 150 nm.

13. The method according to claim 1, wherein the carbon content of the diamond-like solid state material is greater than about 40 atomic % of the DLN, the hydrogen content is up to about 40 atomic % of the carbon, and the sum of the silicon, oxygen and dopants together is greater than about 2 atomic % of the DLN.

14. A cutting apparatus comprising a precision-edged substrate coated with a precision-edge preserving coating, said coating comprising a diamond-like solid state material formed from interpenetrating networks comprising a first diamond-like carbon network stabilized by hydrogen, a second silicon network stabilized by oxygen and, optionally, at least one network of dopant elements, or dopant compounds containing elements from Groups 1–7b and 8 of the periodic table.

15. The apparatus according to claim 14, wherein the carbon, hydrogen, silicon and oxygen are obtained from the decomposition of an organosiloxane having from about 1 to about 10 silicon atoms.

16. The apparatus according to claim 15, wherein the organosiloxane is polyphenylmethylsiloxane.

17. The apparatus according to claim 14, wherein the carbon content of the coating is from about 40 wt. % to about 98 wt. %.

18. The apparatus according to claim 14, wherein the carbon content of the coating is from about 50 wt. % to about 98 wt. %.

19. The apparatus according to claim 14, wherein the carbon to silicon weight ratio of the coating is from about 2:1 to about 8:1.

20. The apparatus according to claim 14, wherein the silicon to oxygen weight ratio of the coating is from about 0.5:1 to about 3:1.

21. The apparatus according to claim 14, wherein the substrate comprises a metal.

22. The apparatus according to claim 14, wherein the substrate comprises a non-metal.

23. The apparatus according to claim 14, wherein the dopant elements are selected from the group consisting of B, Si, Ge, Te, O, Mo, W, Ta, Nb, Pd, Ir, Pt, V, Fe, Co, Mg, Mn, Ni, Ti, Zr, Cr, Re, Hf, Cu, Al, N, Ag, and Au.

24. The apparatus according to claim 14, wherein the carbon content of the solid state material is at least 40 atomic % of the coating, the hydrogen content is up to about 40 atomic % of the carbon, and the sum of the silicon, oxygen and dopants together is greater than about 2 atomic % of the coating.

25. The apparatus according to claim 14, wherein the coating is deposited on the substrate to a thickness of from about 5 nm to about 12 micrometers.

26. The apparatus according to claim 14, wherein the coating is deposited on the substrate to a thickness of from about 5 nm to about 150 nm.

27. The apparatus according to claim 14, wherein the apparatus is selected from the group consisting of industrial tools, surgical instruments, knives and razors.

28. The apparatus according to claim 14, wherein the apparatus is a razor blade.

\* \* \* \* \*



# EXHIBIT D

United States Patent [19]

[11] 3,911,579

Lane et al.

[45] Oct. 14, 1975

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[75]	Inventors: George C. Lane, Danbury; Phyllis M. Curtis, Simsbury; Arthur E. Michael, Middletown, all of Conn.	3,419,414	12/1968	Marks	117/70
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		3,774,703	11/1973	Sanderson	117/75
[73]	Assignee: Warner-Lambert Company, Morris Plains, N.J.				

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[21] Appl. No.: 352,374

Related U.S. Application Data

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[51] Int. Cl.<sup>2</sup> .... B26B 21/54; B21K 11/00

[58] Field of Search ..... 76/104 R, DIG. 8; 30/346.5, 346.53, 346.54, 346.55; 204/192; 117/69 R, 70 C, 71 M, 93.4 R, 107.2 R

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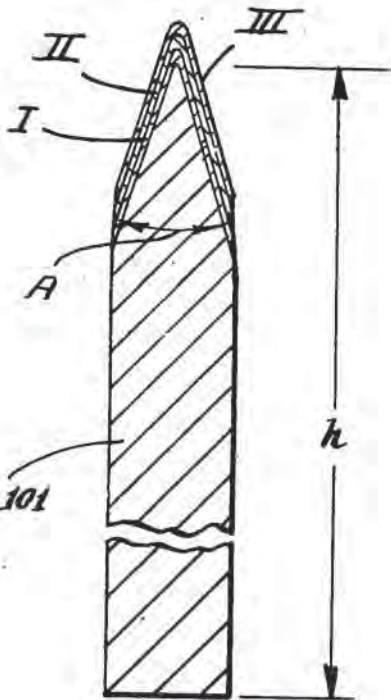
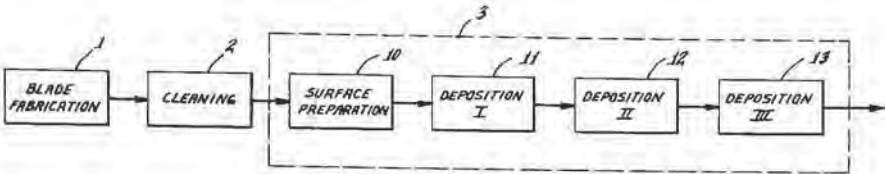
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Primary Examiner—Leonidas Vlachos  
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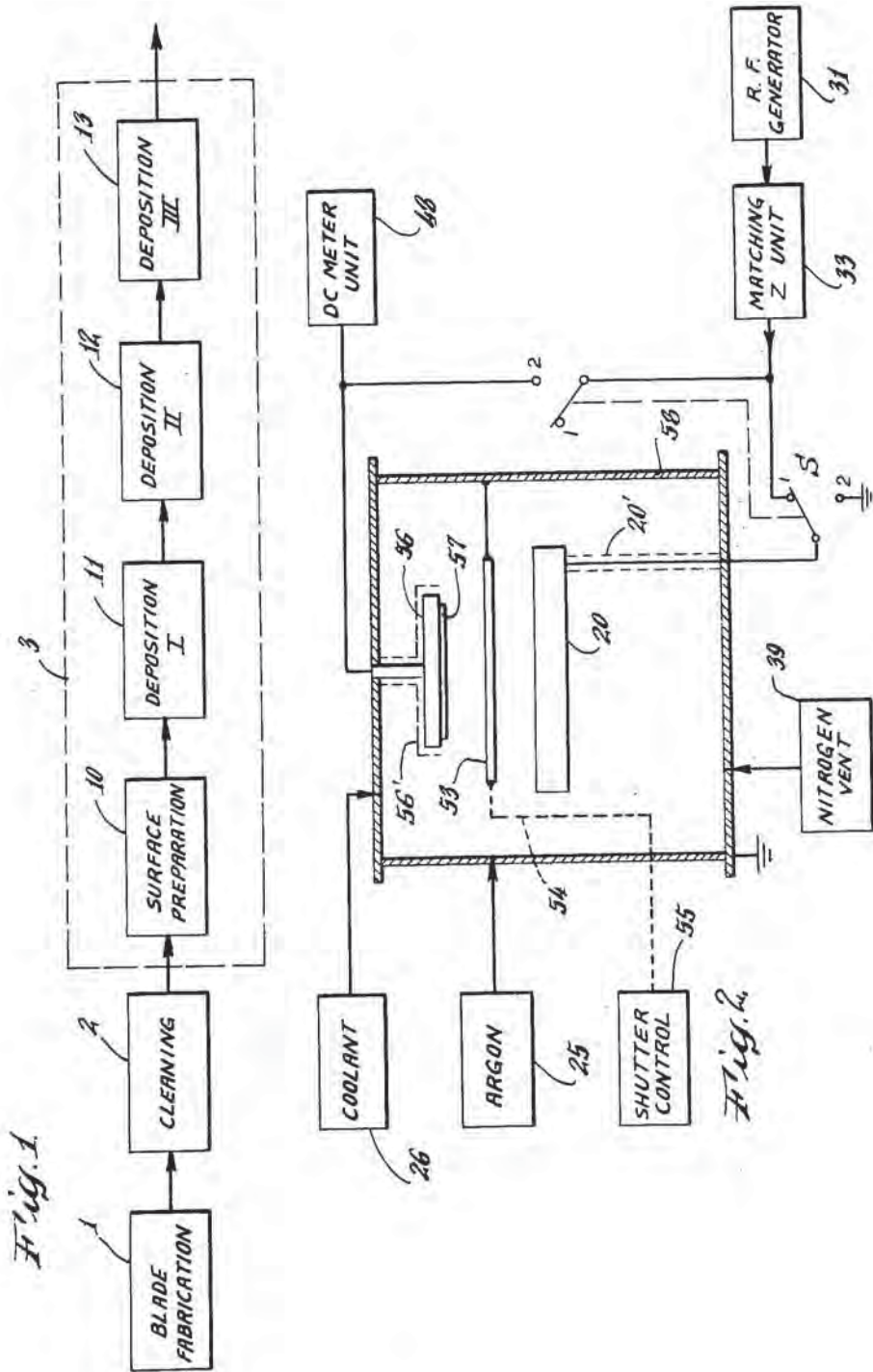
[57] ABSTRACT

The specific disclosure is directed to razor blades and methods of making the same wherein the cutting edge formed by two intersecting surfaces is sputter deposited with a refractory material which is subsequently overlaid with a sputter deposited coating of material displaying adhesion to a final lubricious coating.

66 Claims, 9 Drawing Figures







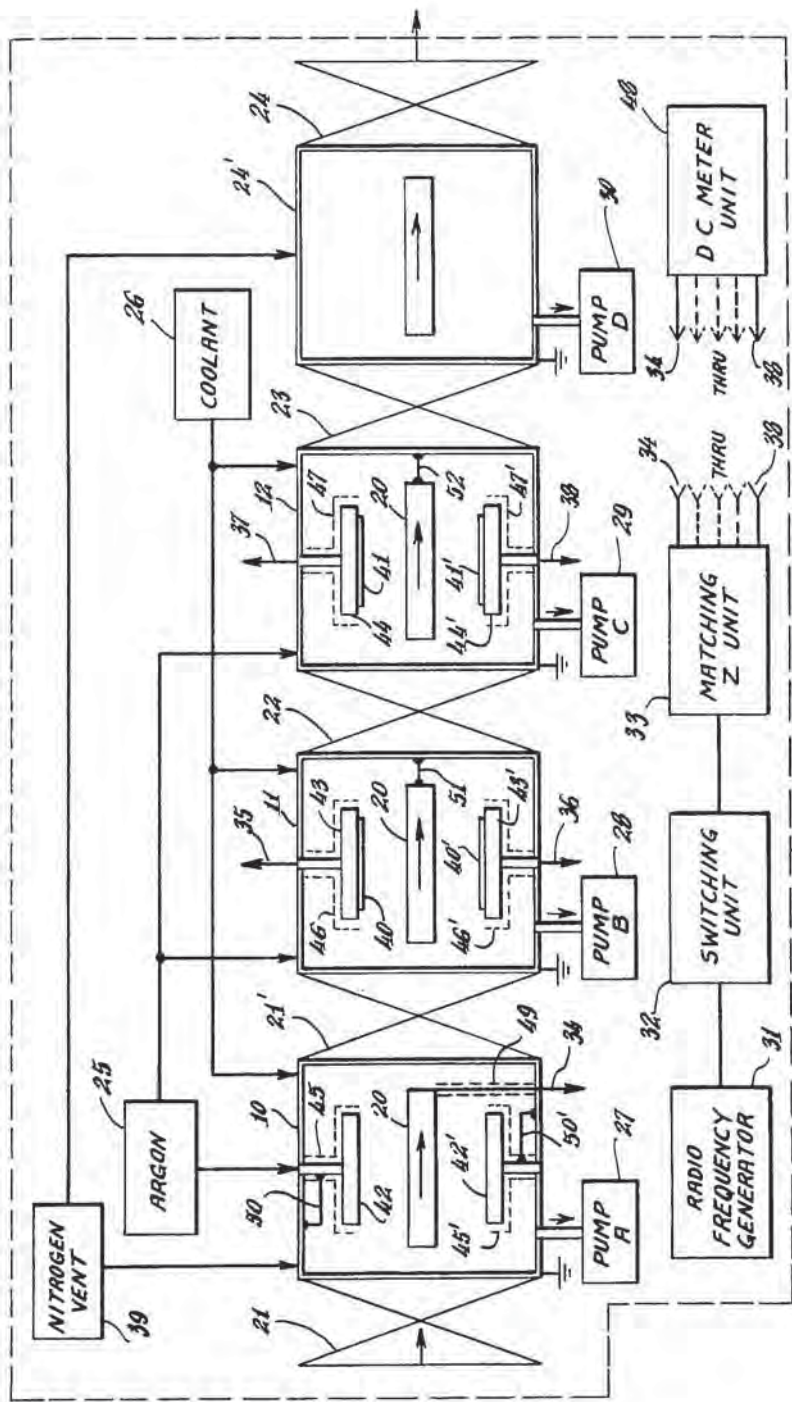
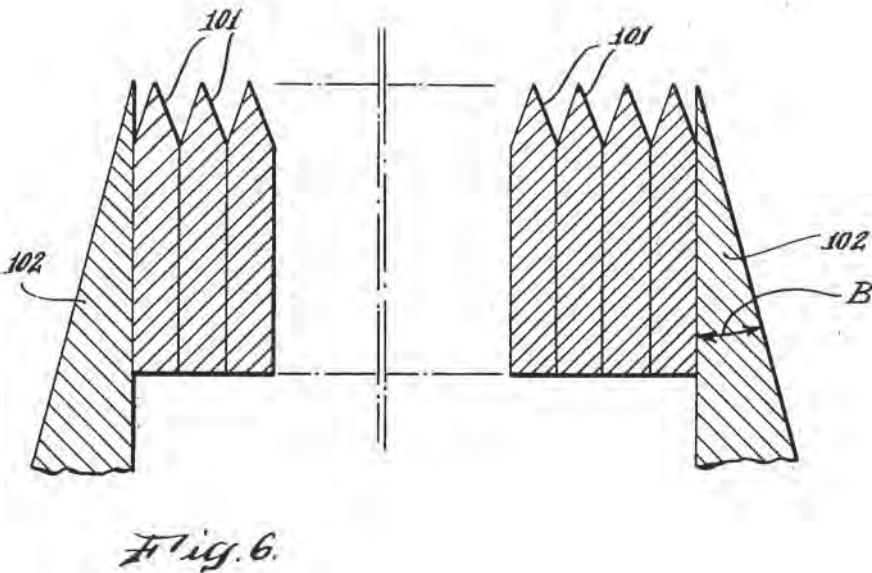
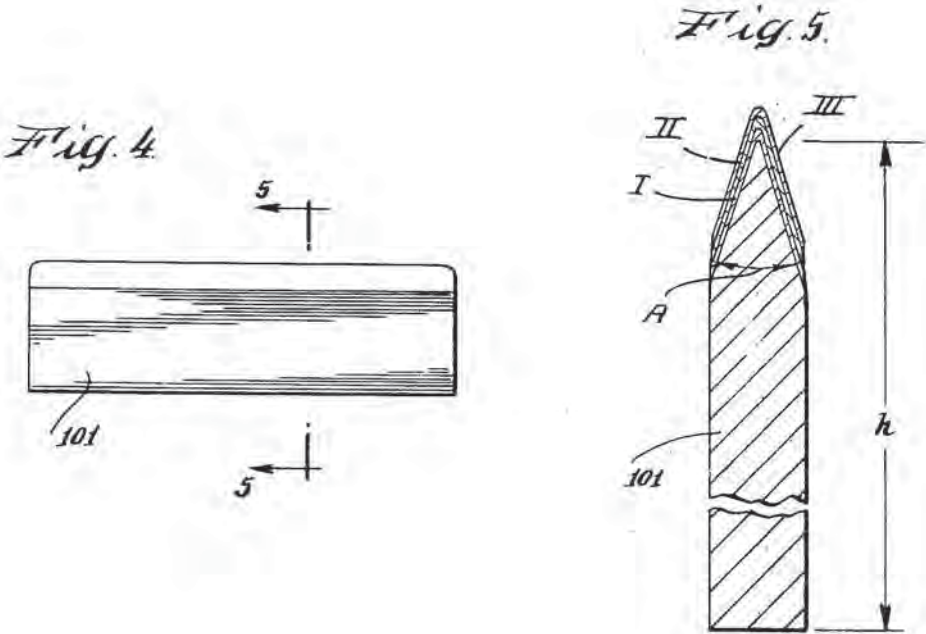
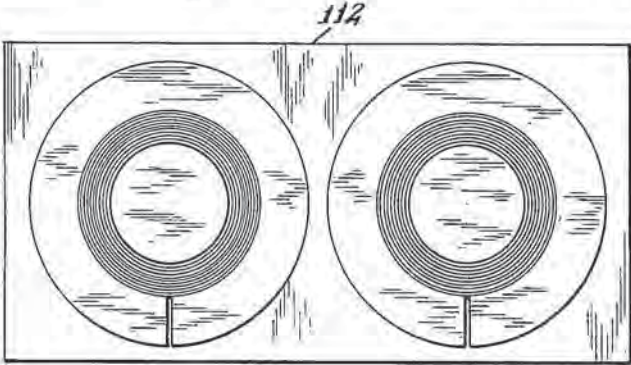
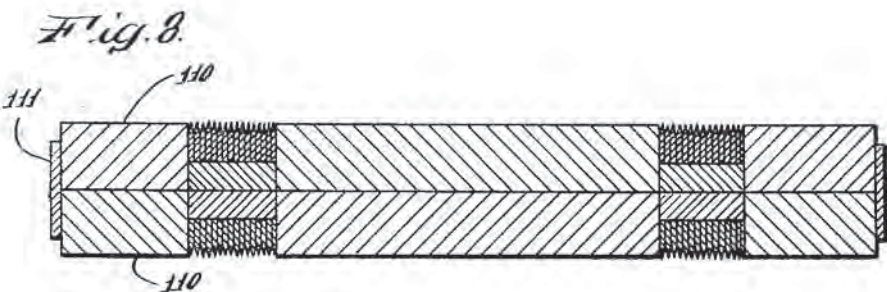
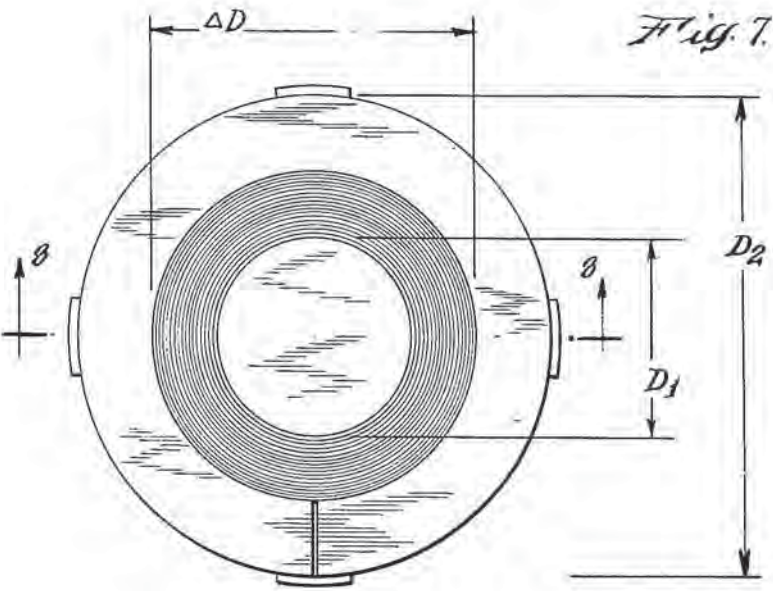


Fig. 3.









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# CUTTING INSTRUMENTS AND METHODS OF MAKING SAME

## CROSS-REFERENCE TO A RELATED APPLICATION

This application is a continuation of U.S. Pat. application Ser. No. 144,509, filed May 18, 1971.

## BACKGROUND OF THE INVENTION

The present invention generally relates to a method for making razor blades and is more particularly directed to a method for producing a razor blade having a cutting edge displaying certain advantages characteristics associated with refractory materials.

The razor blade industry has long sought to produce a product having an extremely sharp cutting edge possessing both long life and concomitantly corrosion resistance. The achievement of these desires has been associated with the producing of a blade made in some fashion from a refractory material. Particular attention has been directed to sapphire or, more broadly speaking, corundum.

Refractory materials by definition comprise various compounds characteristically having a high relative hardness, resistance to working and abrasion under conditions of high temperature, and inertness under most atmospheres and conditions. the making of a razor blade from materials such as these has obvious difficulties. If the refractory material is inherently resistant to working and abrasion, it must, therefore, be extremely difficult to perform the grinding and honing operations necessary to the production of a modern razor blade. It is further characteristic of these materials that they are resistant to bending and thusly do not conform to the strip methods of making blades which are universally in practice today and lead to the economic production of the final product.

Razor blades made from refractory materials, for instance, ceramics, have been extremely difficult to manufacture and, therefore, economically unfeasible under the conditions of today's market. U.S. Pat. No. 3,543,402, R. M. Seager, issued Dec. 1, 1970, entitled "Ceramic Cutting Blade", discloses a method, and the resultant product, for making a razor blade of refractory material. The difficulties involved and the stringent requirements which must be followed are detailed in the specification of this patent and point to its unfeasibility, as previously mentioned. It must be further noted that refractory materials generally do not display the toughness associated with metals, particularly those used in cutting instruments, and their use in view of this is questionable. The orientation of the ceramic crystals and their size become extremely significant when it is realized that the radius of the final apex of most razor blades manufactured today is in the neighborhood of 300 to 500 Angstroms. The abrading or loss of even single crystals from an edge thusly constructed may be of significance to its cutting and life properties.

One of the most significant advances in the art of razor blades has been the use of lubricious coatings applied to the cutting edge. This method of achieving a reduction in the cutting forces involved (shaving comfort) has evolved over a lengthy span of time commencing as far back as the 1930's, and even earlier if one considers the use of shaving lathers in this regard, ultimately resulting in the application of lubricious polymer coatings to the razor blade edges. It may be safely

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said that most razor blades produced today contain a coating of polytetrafluoroethylene (PTFE), which substance has provided an extremely low coefficient of friction and an adherence to the cutting edge commensurate with the ultimate life of the edge itself, i.e., the PTFE appears to remain in operable condition for as long as the blade edge maintains a cutting edge sufficient to sever normal beard hairs. This latter point has been empirically tested and verified through the statistical analysis of data received from extremely large shaving samples.

U.S. Pat. No. 3,518,110, issued June 30, 1970, Inventor: Irwin W. Fischbein, entitled "Razor Blade and Method of Making Same", discloses a method for applying PTFE and like low friction polymeric materials to razor blade edges. This patent does not state the mechanism of PTFE adhesion to the blade but simply hypothesizes that a monolayer of the lubricious material in some fashion, either mechanically or through intermolecular bonding, produces interfacial bonding forces greater than the cohesive forces internal to the coating thereby permitting a minimization of friction and further providing an elimination of asperities between the cutting surface and the material to be severed. Efforts have been made to determine a more exact hypothesis for the apparent improvement in shaving comfort, but, to date, not firm and provable conclusions have been reached. It must be emphasized, however, that the adhesion of the lubricious coating appears to be sufficient to maintain a low coefficient of friction throughout the useful wear life of the blade edge, i.e., blade usefulness is limited by edge breakdown as opposed to loss of lubricity. Experience in the use of razor blade materials other than chromium stainless steel as used in the Fischbein patent has indicated wide variance in the adhesion properties of the lubricious coating; stainless steel and pure chromium and oxides thereof provide extremely long life or adhesion of the coating. Other materials, for instance, platinum and, generally, refractory materials, show a decreased and in some instances no adhesion.

The prior art, although replete with the application of different materials to razor blades, all claimed to improve blade quality and performance in some manner, has generally failed to provide a blade reflecting the overall shaving performance and comfort found in the modern razor blade in combination with the durability of refractory materials as previously discussed. The Seager patent, in addition to the significant problems previously indicated, totally fails to disclose the performance of the claimed blade relative to modern-day products and, in fact, does not show how a final product might be achieved. It is, therefore, an object of this invention to provide a razor blade exhibiting improved qualities.

It is another object of this invention to provide an improved razor blade of a refractory material.

Another object of this invention is to provide a method for applying a refractory material to a razor blade.

Another object of this invention is to provide a method for applying a lubricious material to a razor blade of refractory material.

Yet another object of this invention is to provide a method for depositing a refractory material on a substrate.



Still another object of this invention is to provide a method for depositing corundum onto a substrate.

Still another object of this invention is to provide a method for sputter depositing coatings of material onto a razor blade.

It is yet another object of this invention to provide a method for making razor blades of refractory material in a continuous batch process.

SUMMARY OF INVENTION

In accordance with this invention, a method for making a razor blade is presented. The blade is formed from a suitable material and has an edge portion which consists of two intersecting surfaces which may be honed or made by some other forming process. At least the surfaces comprising the edge as well as the ultimate apex at the intersection are sputter deposited with a refractory material and then, in order to provide adhesion of a final coating of lubricious material, the edge is coated with a second material having the desired adhesive characteristics.

The invention further provides a method of applying an adherent coating of lubricious material to a razor blade edge formed by the intersection of two surfaces of refractory material. This method involves the coating of the refractory surfaces with an overlay of material displaying adhesion to both the surfaces and the lubricious material.

Also in accordance with the invention, there is disclosed a method in which razor blades having edges formed by two intersecting surfaces are sputter etched in a first vacuum chamber. The blades are then moved through a vacuum interlock to a second chamber in which they are sputter deposited with a refractory material. After deposition of the refractory material, the blades are then moved through a second vacuum interlock to a third chamber wherein the coating of material displaying adhesion to a subsequent lubricious coating is sputter deposited on the refractory material. Subsequent to the above steps, the blades are moved through a third vacuum interlock to a fourth vacuum chamber from which they are eventually vented to the atmosphere prior to a final coating with the lubricious material.

Yet another aspect of this invention involves a cutting instrument having an elongate edge of narrow included angle formed by two intersecting surfaces of a refractory material onto which an overlay coating is placed, the overlay coating having adhesion to the final coating of a lubricious material.

The invention is also directed to a method for depositing corundum onto a substrate. This method involves disposing the substrate in an evacuated chamber having an electrode on which is mounted a target of corundum. An ionizable gas is introduced into the chamber and a plasma is established by imposing an RF potential between the electrode and the substrate. Particles dislodged from the target by impingement of gas ions formed in the plasma by the collision of RF excited electrons and the ionized gas are then deposited on the substrate with the needed level of energy to form the desired crystal structure and orientation.

The foregoing summary of the invention as well as other objects and advantages will be made apparent upon a study of the following drawings and the detailed description of preferred and exemplary embodiments.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a functional block diagram of the method and apparatus for producing a razor blade having an edge formed of refractory material.

FIG. 2 is a partially cross-sectional functional schematic showing a typical sputtering chamber.

FIG. 3 is a partially cross-sectional functional schematic showing a multi-chambered continuous batch sputtering system.

FIG. 4 is an outline drawing of a typical single-edge razor blade.

FIG. 5 is a diagrammatic cross-sectional drawing of a typical single-edge razor blade showing in distorted fashion material coatings.

FIG. 6 is a cross-sectional drawing of a batch of razor blades mounted in a holder.

FIG. 7 is a plan view of a fixture for holding a continuous coil of razor blade.

FIG. 8 is a cross-sectional view of FIG. 7.

FIG. 9 is a plan view of a fixture for holding a plurality of razor blade coils.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following detailed description is made in coordination with the drawings of this application and discloses the functional and structural features of the invention. Throughout the drawings and description, conventional symbology and nomenclature is used, and similar units appearing in the different drawings are designated by the same number. It is intended that the descriptions set forth herein be exemplary of the invention and not delimiting of its scope.

A general outline of the steps involved in the manufacturing process for products conforming to the novel features of this application are shown in FIG. 1. As obvious from the nomenclature of the drawing, this process outline is specifically involved with the fabrication of an improved razor blade. The blade is first manufactured in accordance with normal procedures well known in the art. The stainless steel or other applicable blade material is formed into strips of convenient dimension and then passed through punching, heat treating, printing, grinding, and finally honing to produce a final cutting edge formed by the intersection of two surfaces. As previously indicated, this blade fabrication step 1 is amply detailed in the prior art and well known to those skilled in the art. It would serve no useful purpose to go more deeply into the cutting edge manufacture other than to indicate that the different steps involved may be altered to achieve desired characteristics on the edge. To some degree, these achievable characteristics may affect the ultimate process but do not form any intrinsic contribution to the novel concept of the applicants.

After the final edge of the razor blade is formed, which edge is grossly depicted in FIG. 5 of this application and is shown having an included angle A which normally varies between 15° and 25° but may, depending upon the basic substrate or blade material, vary in a much wider degree, it is then passed on to a cleaning step 2. The cleaning step is utilized to remove the contaminants formed on the edge during the fabrication step. Normally these contaminants comprise cutting oils, greases, printing inks, etc., which are a necessary part of the prior process. Again, the prior art quite ade-



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quately documents the type of cleaning needed and the instruments employed to effect its attainment. In a process used by the applicants, the blade edges, while stacked in juxtaposition arrangement, are subjected to jets of trichloroethylene, which fluid is constantly cleaned through a filtration process. Employment of other devices such as ultrasonic energy, agitation, air, etc., are recognized, but, once again, the process used does not lend any novel contribution to the applicants' invention.

The steps depicted in that portion of the process 3 are intimately involved with the applicants' novel process. Contained in this portion of this process are the steps of surface preparation 10, Deposition I, 11, Deposition II, 12, and Deposition III, 13, which are performed sequentially after the cleaning 2. The surface preparation 10 atomically cleans the cutting edge of the blade and prepares it for proper acceptance of subsequent depositions; it normally involves the sputter etching or glow discharge cleaning of the edge, but may be achieved by any process which adequately cleans the intersecting surfaces forming the edge by the atomic removal of blade substrate material, contaminants and adsorbed gases. The description of the invention hereinafter presented in the body of this application will describe those surface preparations believed most adaptable to the inventive method of the applicants, but it must be recognized that this preparation may vary without departing from the scope of the applicants' novel contribution. Deposition I coats the blade edge formed by the intersecting surfaces with a refractory material by a suitable deposition process. In that refractory materials are generally of a dielectric nature and since further radio frequency sputtering has achieved desirable performance results, this first coating of refractory material is applied by an RF sputtering process. Again, however, it must be recognized that any sputtering process, whether it be alternating current of sufficiently high frequency or a modified direct current sputter process having means for dissipating the charge sheath formed about the cathode or variant forms of bias sputtering, may be utilized. The nature and apparatus of sputtering is adequately set forth in Chapter 3, pp. 3-2 through 3-35, in "Handbook of Thin Film Technology" edited by Leon I. Maissel and Reinhard Glang, published by McGraw-Hill Book Company, 1970. It is significant to point out at this juncture that the nature of the deposition process is not necessarily significant to the invention as long as it achieves a satisfactorily adherent and continuous coating of the refractory material which at this time can, within the applicants' knowledge, only be achieved by a sputtering or equivalent process.

The refractory material to which greatest attention is currently being directed in the application of this invention is synthetic sapphire or, as previously indicated, corundum. This material when sputtered on the edge of a razor blade clearly displays the characteristics previously set forth herein which are desirable and necessary to the production of an improved razor blade. It must be noted, however, that other generally classified refractory materials such as glass, quartz, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride amongst others may be successfully used in razor blade or cutting edge applications. It must be further recognized that this invention is not necessarily limited to refractory materials but may find equal application

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to any material displaying desirable blade or cutting edge characteristics without having the necessary or preferred degree of adhesion to a subsequent lubricious coating. Further, it is significant to point out that in sputter depositing a preferred crystalline structure of aluminum oxide, an aluminum target may be used with a reactive oxygen containing atmosphere. With appropriate choice of operating parameters and oxygen, a desired morphology and composition may be deposited.

Deposition II constitutes the coating of the blade edge with the material displaying the desirable adhesion to the subsequent coating of lubricious material. Rather immense statistical evidence has indicated a superior degree of performance by the use of preferably chromium or some chromium alloy coatings. This material not only adheres strongly to the lubricious coating but provides a hard and durable shaving edge. In addition to chromium, other materials, namely, platinum, aluminum, titanium and iron, amongst others, and alloys of these metals, have found application to razor blade edge coatings. As our knowledge of the mechanics of adhesion increases, it may very well develop that other materials we well as the one mentioned herein may find application within the scope of this invention. The preferred method of deposition involves the radio frequency sputter depositing of chromium onto the blade edge. Of course, DC sputter coating may be used to apply the second material to the razor blade, but it has been determined that in overall aspect the use of RF sputtering techniques seems to lend a decided improvement to the product. It is generally hypothesized that this improvement is to some degree due to the inherent cleaning and desorption of gases on the surface of the razor blade edge which takes place during the RF sputtering process. The second material must be deposited to a thickness sufficient to provide the desired degree of adhesion to the subsequent lubricious material. It has been found that this desired characteristic is achieved by applying a coating of approximately 25 Angstrom units in thickness. The performance of such a thin coating is extremely surprising in that it forms only approximately a coating of five atomic layers in thickness and further cannot be considered continuous over the entire surface of the refractory material. It is pointed out, however, that this material may be deposited to any thickness sufficient to provide the desired adhesion limited only by the requirement that the thickness not in any way detract from the sharpness of the cutting edge. It has been found that thicknesses of up to and greater than approximately 300 Angstroms are completely compatible with the cutting properties of the blade edge.

It is not understood why such an extremely thin coating of material produces such remarkable improvements in adhesion of the lubricious coating. It is hypothesized that the second material provides a desirable crystal or other surface morphology to which the polytetrafluoroethylene or other lubricious material may find favorable adhesion through mechanical locking of the surfaces. Although such hypothesis seems totally acceptable for thicker films, it can be reasonably questioned when considered with films as thin as 25 Angstroms. In this regard, it has been theorized that perhaps the thin chromium or other material coating alters the surface energies of the material in such a manner as to permit some form of energetic linking be-



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tween the molecules or atoms of the polymeric coating and the overlay coating of chromium either alone or in combination with the first coating of refractory material. The applicants, however, do not wish to be limited to the mechanism of adhesion achieved in the practice of this invention, but rather simply use the ultimate fact of its performance within the context of their novel contribution.

Deposition III involves the final process step for coating the razor blade edge with a lubricious material. As previously set forth, this coating of lubricious material is generally considered as necessary for the proper performance of all razor blades manufactured today. The Fischbein patent, *supra*, adequately describes methods for applying a coating of polytetrafluoroethylene, which method is wholly compatible with the novel process of the applicants. Briefly, after the Deposition II coating is applied, the blades are in stacked alignment sprayed with an aqueous or Freon based dispersion of low molecular weight polytetrafluoroethylene, the thickness of such coating being substantially greater than 2,000 Angstroms. After spraying, the blades are then subjected to a heat somewhere in the range in excess of 600° F. for a limited period of time. During this heating process, the blades are maintained in a substantially inert environment comprising nitrogen or cracked ammonia. It is, however, pointed out that certain more reactive gases may be added to the environment or ambient conditions of the blade during heating to provide certain desired characteristics such as improved adhesion of the polytetrafluoroethylene. The improvements in adhesion lent by the variations in the heating atmospheres, however, are not considered as part of the applicants' invention and are generally considered negligible with respect to the improvement in adhesion provided by Deposition II. In addition to polytetrafluoroethylene, other polymeric materials have found some application to razor blade edges, although up to this time not providing the same degree of performance as polytetrafluoroethylene. These are polypropylene, polyhexafluoropropylene, polychlorotrifluoroethylene and polyethylene, amongst others. It is entirely conceivable that the polymers mentioned, as well as others not presently considered for use, may find future application to razor blade edges if the necessary modifications to the process to achieve desirable performance are discovered or if the polymer molecules are in some manner modified or cross-linked to alter their characteristics in an advantageous manner.

FIG. 2 presents in schematic outline form a partial cross-section of a vacuum chamber 58 associated with ancillary equipment without the chamber 58 and internal appendages within the chamber 58 necessary to the performance of the applicants' method. Within the chamber there is diagrammatically presented an RF electrode 56 surrounded by a shield 56' necessary to prevent leakage of RF to its surrounding environment. On the face of the electrode there is located a target 57 which, depending upon the step of the process being performed, may comprise the refractory material, Deposition I; or the Deposition II material, namely, chromium, or other material as previously mentioned. The target 57 may be cemented to the face of the electrode or preferably mounted to the electrode through screws or other fastening devices which do not extend to the front of the target thereby preventing any contamination

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tion of the target or the substrate to be sputtered by the material of which the fastening devices are made. The electrode 56 is brought by means of suitable RF insulators and couplers to the outside of the chamber for its connection to the source of energy. The second electrode 20 comprises a member which includes the blade or blades or other devices on which material from the target 57 is to be sputter deposited. An RF lead is brought from the substrate through the wall of the chamber by means of suitable RF connectors and couplers to the outside source of energy. Similarly to the electrode 56, an RF shield 20' is provided to prevent leakage of energy to the surrounding environment.

Movement of the shutter 53 is provided by a mechanical linkage 54 brought through the walls of the vacuum chamber 58 to a control unit 55. This mechanical drive train 54 which may comprise any suitable mechanical linkage, for example, driven gear systems or flexible shafts or rack and pinion or screw rod drives, is provided with the necessary driving force by the control unit 55 which may comprise any suitable AC or DC motor drive limited by microswitches within the chamber sensing the position of the shutter 53. Of course, the passage of the mechanical linkages through the vacuum chamber 58 must be adequately sealed by O Ring configurations, bellows or other suitable sealing members.

A coolant unit 26 normally comprises a pump or pressure line for forcing water or other suitable coolant through passages provided within the electrode 56. In the sputtering process significant heat is generated in the electrode 56 and generally it is found advantageous, if not always necessary, to provide some medium for heat transfer from the electrode 56 in combination with the target 57 in order to prevent burnout of the sputter electrode configuration. Of course, any other members which may during any particular process require coolant may be provided with such medium by the same coolant unit 26. During the sputtering process, it is necessary to generate an electrical plasma. This plasma is maintained by the presence of an ionizable gas. In the present invention, Argon is found most suitable and is provided to the chamber by the Argon unit 25. Suitable valving for admitting the desired amount of Argon is well known to those skilled in the art and may provide a needle valve arrangement of rather simple construction. The nitrogen vent unit 39, similar to the Argon unit 25, provides for the admission of a gas to the inside of the vacuum chamber 58. The nitrogen vent 39 serves two purposes to the apparatus shown in FIG. 2. Firstly, it permits purging of the internal space of the chamber 58 prior to commencement of the steps of the process, thereby providing a drying and cleaning action to the chamber 58. Secondly, the nitrogen vent unit 39 provides through a suitable needle valve or other arrangement for the admission of gas to the chamber 58 prior to the opening of the chamber upon completion of a process step, thereby preventing potential damage to the equipment as well as the seals, etc., associated with the equipment which may be attendant to the sudden loss of vacuum. Further, it may be extremely difficult to open various parts of the chamber 58 without a reduction in vacuum provided by the nitrogen vent 39. It is pointed out that the coolant unit 26, the Argon unit 25, and nitrogen vent unit 39 must all be adequately sealed to prevent leakage within the chamber 58 environment.



A DC meter unit 48 provides a means for measuring the bias voltage which is built up on the electrode 56 during sputtering operations. This bias voltage is normally considered a figure of merit with respect to the degree of sputtering or sputtering rate which is preferred during the coating process. The RF generator 31 provides the energy source for the radio frequency sputtering operation. Generally, in conformance with FCC Regulations, a frequency of 13.56 megacycles is used. It must be pointed out and recognized, however, that any suitable high frequency may be employed notwithstanding FCC Regulations. The matching Z unit or matching impedance unit 33 provides for proper power matching or impedance matching of the RF generator 31 and the input to the RF electrode 56. This impedance viewed looking into the electrode 56 is a complex affair determined not only by the configuration of the electrodes internal to the chamber 58 but by the operation of the plasma generated during the sputtering cycle. This matching unit 33 comprises normally various inductive and capacitive components in pi, T and series or parallel arrangements necessary to achieving certain impedance matching. A copending patent application of one of the applicants, viz., U.S. Ser. No. 680,926, filed Nov. 6, 1967, now U.S. Pat. No. 3,632,494, dated Jan. 4, 1972, shows one matching unit 33 configuration which may be employed with the device shown in FIG. 2 or with similarly arranged sputtering equipment. It must be realized, however, that the establishing of the matching unit 33 parameters is substantially an empirical process varying to some degree with the particular sputtering equipment being utilized. It is considered that the design and determination of the matching unit 33 configuration is well within that level of knowledge commensurate to those individuals ordinarily skilled in the art.

The switch S provides for altering the connections from the electrodes to the ground of the system and to the matching unit 33 for achieving the different steps of the sputtering process. This switch S may necessarily be ganged with other switches or switch in the matching unit 33 to alter the output impedance configuration to conform with the changed impedance level when the switch S is moved to its second position. In position 1, it is obvious that the RF energy is applied to the substrate electrode 20. In this circuit configuration, a plasma is formed which generates a sputtering action by attracting positive Argon ions toward the substrate electrode 20. This attraction is mainly provided by the buildup of a negative bias voltage on the electrode 20 which results essentially from a series capacitor in the line between the substrate electrode 20 and the matching unit 33. This capacitor is normally provided within the matching unit 33. This configuration thus satisfies the requirements of the surface preparation step 10 by atomically cleaning the edge of the blades or other cutting instruments. Naturally, the rate of material removal must be carefully and closely maintained.

When switch S is placed in position 2, the substrate electrode 20 is brought to system ground as is the chamber 58 while the RF energy is connected through the matching unit 33 to the electrode 56. When in this configuration, two desirable results may be achieved. With the shutter S3 interposed between the substrate electrode 20 and the RF electrode 56, the buildup of the plasma within the chamber 58 causes sputtering of material from the target 57. This material, however,

cannot impinge upon the substrate 20 due to the interposition of the shutter 53 thereby cleaning the target surface 57 prior to the deposition of any material onto the substrate electrode 20. Once the shutter 53 is removed by the coaction of control unit 55 drive linkage 54, continued application of RF energy brings about the sputter deposition of target material onto the substrate surface or, in this case, the cutting edges of the razor blades. As is obvious from the mechanical configuration of the sputtering apparatus, the chamber must be opened between steps of Deposition I and Deposition II. The shortcomings presented by this necessitated opening and re-evacuating of the chamber 58 are overcome by equipment conforming essentially to that presented in FIG. 3 of this application. There is shown in FIG. 3 a continuous batch sputtering process apparatus which is uniquely adaptable to the applicants' invention. Prior, however, to discussing the operation of this apparatus within the context of the applicants' process, operation of the equipment as well as the steps of the applicants' novel process will be considered with respect to the equipment of FIG. 2.

In considering an operational analysis of the equipment and the process involved, it may first be beneficial to consider the blade holder configuration presented in FIG. 6. FIG. 6 shows in distorted dimension a partial cross-sectioning of a typical blade stack held transversely by the blade holder. It has been found for reasons not wholly understood that in order to achieve the desired uniformity of sputter deposition coating both across the individual blades as well as throughout the entire stack of blades 101 the geometric configuration of the holder ends 102 is extremely important. The apex of the end portions 102 must lie in substantially the same plane as the apex of the blade 101 contained within the holder structure. It has been discovered that the maintenance of a maximum fall off angle from this apex is essential or, speaking in complementary terms, the included angle B of end members 102 of FIG. 6 must be held to a minimum compatible with the strength necessary to apply the compressive forces to hold the blades in proper alignment.

It has been found empirically that, using a 304 type stainless steel blade holder, this angle may be successfully limited to approximately 15°. It is hypothesized, however, that even a smaller angle may be successfully used if suitable material is utilized. However, the 15° angle is found to supply satisfactory performance as well as an acceptable life during commercial deployment of the equipment. As previously indicated, the effects of the geometric configuration of the holder are not understood. However, it is recognized that any modification of electrode 20 configuration will alter the shape and potential of the plasma and thereby affect the distribution and energies of the sputtered target 57 material. The transverse sides or sidewalls of the holder are not presented in that no particular configuration seems to be needed except that the plane of these sides must fall substantially in the plane of the apex of the blades 101 and extend for some reasonable distance around the periphery of the blade 101 stack. It is significant to point out particularly with regard to the equipment of FIG. 3 wherein dual facing electrode configurations are employed that a complementary form of the blade holder shown in 102 may be provided to stack a second set of single-edge blades 101 in contraposition to that shown in FIG. 6, thereby providing



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for contemporaneous sputtering of both edges as the holder is disposed between oppositely facing target electrode combinations. Similarly a blade holder of substantially the same geometric configuration with the bottom planar member eliminated may be used to hold double-edge blades for simultaneous sputtering of both edges in the same dual electrode configuration.

Returning now to the novel process of the applicants and its performance within the confines and context of the sputtering equipment of FIG. 2, the razor blade, after cleaning in conformance with the process step 2 of FIG. 1, and it might be noted that as soon after this step as possible, the blades are disposed either singly or in stacked arrangement as shown in FIG. 6 within the vacuum chamber 58 and rigidly attached to the substrate electrode 20, thereby forming a part of such substrate electrode 20. The target material 57 required for Deposition I is then adequately fixed to the surface of the electrode 56. Upon sealing of the vacuum chamber 58 and subsequent to its purging by nitrogen through the nitrogen vent unit 39, the chamber is evacuated by a suitable pump configuration (not shown). The pumping configuration may comprise mechanical roughing pumps for first reducing the internal pressure of the chamber 58 to the range of  $10^{-3}$  Torr and in addition might then include turbomolecular pumps, diffusion pumps, ion pumps or cryo-pumps together or in combination to further reduce the internal working pressure of the chamber 58 to a level of approximately  $10^{-6}$  Torr, which, under normal circumstances, is considered compatible with RF or in fact most sputtering processes. Upon reaching the desired level of vacuum as previously indicated, approximately  $10^{-6}$  Torr, Argon is admitted to the chamber thereby lowering the vacuum level to between approximately  $1 - 2 (10)^{-3}$  Torr. With the application of RF energy through the RF generator 31 and the matching unit 33 and the switch S position 1, a plasma is established between the electrodes and a negative self bias forms on the substrate electrode 20. Once the plasma has formed, the energetic collisions of the plasma electrons with the Argon gas molecules causes the formation of positive Argon ions which, as previously indicated in this application, are attracted toward the surface of the substrate electrode 20. Upon their impingement on the blade edges, the apex of which are disposed toward the electrode 56, there is a resultant dislodgement of material both blade steel as well as contaminants thereon from the blade edges. This process is continued for a predetermined period of time commensurate with operational conditions determined to some extent by the blade material utilized and the cleanliness of the blade edges after the cleaning process step 2. Typical ranges of time for the completion of this, what is known as etching or sputter etching, process varies between 3 and 10 minutes. Other typical values involved in this sputter etching step is the application of between 300 and 800 W. of RF power with approximately zero refracted power and the development of approximately a thousand volt or 1 KVDC self bias on the substrate electrode 20. Of course, during this time, the coolant unit 26 is maintaining the electrodes at a desirable temperature compatible with the material used and the allowable range of temperature within the vacuum chamber 58.

After performing this sputter etching of the blade holder razor blade combination located on the substrate electrode 20, the switch S is moved to its second

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position in which the RF energy is applied to the electrode 56 while the substrate electrode 20 is connected to system ground. Again, with the shutter still maintained in its interposed position between the electrodes 56, 20, the plasma is again established at an Argon or vacuum pressure of normally higher value, typically between 5 and 7  $(10)^{-3}$  Torr. The power level is raised to a higher value, typically in the range of 1.5 KW of real power, and the bias reaches a considerably higher level, typically in the range of 2 KVDC negative. In this circuit configuration, the development of a negative self bias voltage on the electrode 56 now attracts the positive Argon ions generated within the plasma toward the target 57, resulting in a sputtering of material, both target material and contamination from the surface of the target 57. This results in a pre-cleaning of the target prior to performance of the actual sputter deposition process onto the razor blades contained in the substrate electrode 20. Typically this pre-cleaning operation is continued for a short interval of approximately 1 minute. Further pre-cleaning or pre-cleaning for a longer time is not normally considered necessary when the target material is substantially pure and kept in a clean environment.

With proper manipulation of the control unit 55, the shutter is then removed from between the electrodes contained in the vacuum chamber 58, thereby exposing the surface of the target 57 to the apex of the blades disposed on the substrate electrode 20. At this juncture, the material sputtered from the surface of the target 57 is allowed to impinge upon and coat the razor blade edges properly disposed toward the electrode 56. Since the essential configuration of the internal vacuum chamber circuitry is not altered or greatly altered by the removal of the shutter 53, the working parameters of the sputter deposition step remain essentially the same as that of the pre-clean step, i.e., the pressure level is maintained in a range approximately  $5 - 7 (10)^{-3}$  Torr, the real RF power is approximately 1.5 KW, and the self bias negative voltage developed on the electrode 56 is approximately 2 KVDC. The period of sputter deposition is, of course, varied greatly depending upon the material forming the target 57 as well as the desired thickness of coating. Many materials display widely variant sputtering rates depending upon the structure or morphology of the target 57 as well as the work function of the material utilized. Typically depending upon the target 57 material, the sputter time may vary between 1 and 15 minutes.

The chamber 58 is vented by the nitrogen vent unit 39 by the admission of nitrogen gas to the chamber through a needle valve connection. Once the chamber is vented to approximately atmospheric pressure, the chamber 58 is then opened and the target material 57 is changed to the material which is to be used in the Deposition II step. As previously indicated, the preferred material is a pure chromium. Normally the purity of this chromium target is maintained in excess of 99.99 percent. However, less pure targets may be utilized without detracting from the quality of performance and the results of the novel process of the applicants. The new target material 57 is attached to the electrode 56 in the same manner as the previous refractory target material for the Deposition I step. It may well be timely to point out that a chamber 58 constructed to have more than one electrode target configuration may be constructed to perform the process outlined herein. If



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dual targets were arranged and could be properly indexed, the subsequent coating applied in the Deposition II step may be deposited without the need for opening the chamber and changing the target material. It is also noted that the substrate and shutter combination 20 and 53 respectively may be indexed to a different location and placed thereby under a different target material as opposed to indexing or changing the electrode configuration which may cause some problems or difficulties associated with the RF connections.

After changing the target material to chromium, the chamber 58 is again sealed and the steps preparatory to sputter deposition are repeated with the exception of the surface preparation 10 step, i.e., the sputter etching of the substrate 20 prior to sputter deposition. Obviously, the surface coating applied during the sputter Deposition I step need not be sputter etched prior to a subsequent deposition in that the material laid down on the surface is substantially pure and free of contamination. Briefly, the chamber 58 is purged by dry nitrogen supplied to the chamber 58 by the nitrogen vent unit 39; the pump unit 58' then evacuates the chamber to a level of approximately  $10^{-6}$  Torr; Argon is then admitted to the chamber 58 through the Argon 25 unit to a pressure of between  $5 - 7 (10)^{-3}$  Torr and, of course, the coolant unit 26 continues to supply a cooling fluid through the appropriate RF electrodes. With switch S in position 2, RF energy is applied to the electrode 56 with the shutter 53 disposed between the substrate 20 and the electrode. The power levels are adjusted to approximately 1.4 KW and the self bias voltage developed is approximately 2 KVDC. Using these operative parameters, the new target 57 is pre-cleaned for a period of approximately 1 minute.

After completion of the pre-clean step, the target 57 is exposed to the substrate 20 by removal of the shutter 53 through operation of the shutter control unit 55. With the vacuum level in a range between  $5-8 (10)^{-3}$  Torr, the substrate 20 is then sputter deposited with the target 57 chromium material for approximately 1 minute. Under these controlling conditions, a coating of chromium approximately 25 Angstrom units in thickness is applied to the substrate 20 principally falling upon or impinging upon the cutting edges of the blades contained integrally within the substrate electrode 20. As heretofore indicated, this period of sputtering may be prolonged for a greater time if a thicker coating of chromium material is desired to the extent that such thickness dimension is compatible with the desired cutting edge sharpness of the blade which is determined through various test equipment well known to those ordinarily skilled in the razor blade art. One well established test for the determination of sharpness is the cutting of nylon fibers disposed on a moving belt at a certain angle to the razor blade cutting edge. A measurement of the cutting forces involved in this test provides acceptable data to a determination and correlation of edge sharpness. Normally, or at least under most circumstances, the final radius of curvature of the cutting edge of a razor blade is approximately in the 400 Angstrom range. This radius provides a relative indication of the allowable total thickness of the material provided by the combination of the sputter Deposition I step and the sputter Deposition II step. Certainly, greater than a total thickness in the range of 500 Angstrom units may not be acceptable.

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After completion of the Deposition II step, the chamber 58 is again vented through the nitrogen vent unit 39 and opened to the atmosphere for removal of the blades. The blades are then spray coated or otherwise coated with the appropriate lubricious material, normally polytetrafluoroethylene, and subjected to the thermal process necessary to provide a final adherent coating. This thermal process mainly involves the evolving from the dispersion of the volatile mediums necessary to the application of the PTFE constituents. The temperature of the heating process in addition to boiling off or evaporating the volatile medium raises the PTFE dispersed particles to approximately their fusion temperature so that in essence the PTFE is sintered to the surface forming an approximately continuous coating over the ultimate apex of the razor blade edge and the facets or intersecting surfaces forming such apex.

FIG. 5 demonstrates in typical cross-section a razor blade of distorted dimension and form showing the final product having thereon the material coatings applied during Deposition I, II and III steps. The blade is of the single-edge type having a height from base to ultimate apex or cutting edge of H. The cutting edge is shown as formed by two intersecting surfaces having an included angle A therebetween. In accordance with products actually sold and used today, these intersecting surfaces actually comprise a number of facets having different included angles, only the final facets having the same angle A as depicted for the intersecting surfaces of FIG. 5. Normally, all the facets and to some extent the body of the blade 101 is covered with the various coatings comprising the novel process of this application. However, it is not necessary for performance that these coatings do extend beyond the facets of the blade.

The first coating applied to the blade designated as I conforms to the material applied or deposited during the Deposition I step. This sputter deposited coating is normally the refractory material previously mentioned or as also indicated some other material which may have desirable blade characteristics but which does not have the ultimate adherence to PTFE coating. The thickness of this coating as it wraps about the ultimate edge of the blade is usually chosen to be between 200 and 300 Angstroms, appreciating, however, that this thickness may be radically changed if different blade edge characteristics are desired, such as increased or decreased blade sharpness. The coating designated as II correlates with the Deposition II step and as indicated normally is a chromium coating. Although this coating is shown as having essentially the same thickness as I and II coatings, it is noted that this thickness is normally in the range of 25 Angstrom units which, on a relative scale, would be impossible to show within the drawing of FIG. 5. Thus, for demonstration purposes, the same thickness coating is shown. The III coating is that placed on the blade during the Deposition III step. As indicated, normally this coating is applied by a spray with subsequent heating for formation of a substantially continuous and uniform coating. However, as indicated in copending Application Ser. No. 680,794, filed Nov. 6, 1967, now U.S. Pat. No. 3,635,811, dated Jan. 18, 1972, this final lubricious coating may also be applied by a sputtering process which may be performed in the same chamber 58 and with the same equipment as shown in FIG. 2. Of course, if such final lubricious



coating is to be sputtered, the target material 57 as well as the operating parameters of the chamber must be significantly modified. Since this final lubricious coating III is of greatly increased thickness in the range of 2,000 Angstrom units and considerably higher, its thickness as shown in FIG. 5 is greatly distorted in order to show the coating without having to scale the razor blade and coatings I and II to relative dimensions not capable of demonstrating the points of most interest with respect to the conformation of the final product. FIG. 4 shows a plan view of blade 101 indicating that the coatings extend substantially continuously throughout the entire expanse of the final facets of the razor blade edge.

To better demonstrate the applicability of the novel process presented herein and to provide a clearer understanding of both the equipment and the various steps employed, the following examples are presented:

EXAMPLE 1

Standard double-edge stainless steel razor blades of approximately 0.004 inch thickness were cleaned in accordance with the cleaning step 2 and mounted within a vacuum chamber substantially conforming to that depicted in FIG. 2, and this example and the following examples will be discussed in the context of the equipment as shown in FIG. 2. A single edge of the double-edge blades are disposed on the substrate electrode 20 in facing relationship to electrode 56 and the target 57. The remaining parameters of this example will be presented in outline form in accordance with the steps heretofore presented:

Surface preparation step 10	
Initial background vacuum	10 <sup>-6</sup> Torr
Argon pressure	1 - 2 (10) <sup>-6</sup> Torr
RF power	400 W.
Self bias voltage	1 KVDC
Sputter time	5 minutes
Target 57 pre-clean step	
Argon pressure	between 5 - 7 (10) <sup>-3</sup> Torr
RF power	1.4 KW
Self bias voltage	2.2 KVDC
Sputter Deposition I	
Target 57 material	Linde synthetic sapphire comprising essentially hexagonal crystal lattice structures of AL <sub>2</sub> O <sub>3</sub> manufactured by the Linde Crystal Products Division of Union Carbide 4" in diameter by ¼" thick
Target dimensions	between 5 - 7 (10) <sup>-3</sup> Torr
Argon pressure	1.4 KW
RF power	2.2 KVDC
Self bias voltage	approximately 7-½ min.
Sputter time	approximately 30 Angstroms per min.
Sputtering rate	between 200 and 300 Angstrom units
Coating thickness	
Sputter Deposition II	Pure chromium
Target 57 material	
Target pre-clean step	between 5 - 7 (10) <sup>-3</sup> Torr
Argon pressure	1.4 KW
RF power	2.2 KVDC
Self bias electrode voltage	1 minute
Period	
Sputter deposition step	between 5 - 7 (10) <sup>-3</sup> Torr
Argon pressure	1 KW
Power	2.2 KVDC
Self bias DC voltage	10 seconds
Time	approximately 180 Angstrom units per min.
Sputter rate	approximately 30 Angstrom units
Coating thickness	

Thereafter Deposition III step was performed and a coating of PTFE was applied to the blade surface. Standard tests showed the blade to display a low coefficient friction and an increased wear life.

EXAMPLE 2

The conditions of Example 1 were repeated in Example 2 with the exception of the sputter etch time, which was reduced from a 5-minute interval to a 1-minute interval. Identical results were obtained with regard to performance of the ultimate product after application of the final lubricious coating of PTFE.

EXAMPLE 3

The equipment was set up in the same manner as Examples 1 and 2. The blade edge was sputter etched under the following conditions:

RF power	200 W.
Argon pressure	1 (10) <sup>-3</sup> Torr
Self bias voltage	1 KVDC
Sputter etch time	5 min.
Target pre-clean step	
Target material	quartz
Argon pressure	Same as Example 1
RF power	Same as Example 1
Pre-clean time	Same as Example 1
Self bias voltage	Same as Example 1
Sputter Deposition I step	
Argon pressure	Same as Example 1
RF power	Same as Example 1
Time	Same as Example 1
Self bias voltage	Same as Example 1
Thickness	approximately 200 Angstrom units
Sputter Deposition II step	
Target material	Pure chromium
Pre-clean conditions	Same as that for pre-cleaning of the Deposition I target
Sputter deposition step	
Argon pressure	Same as Example 1
RF power	Same as Example 1
Sputter time	Same as Example 1
Self bias voltage	Same as Example 1
Sputter coating thickness	approximately 30 Angstrom units

The blade of this example performed in a similar manner to that produced under Examples 1 and 2 and similarly displayed improved friction and life characteristics. Note: Throughout Examples 1 - 3, a Bendix 6 inches diffusion pump system with a liquid nitrogen baffle was used to produce the desired vacuum levels, and throughout the three examples the distance from the target to the blades was approximately 2 inches.

As clearly demonstrated by the foregoing disclosure, razor blades displaying improved shaving characteristics may be produced by the outlined methods and the indicated examples. Processes performed in conformance with the foregoing teaching will produce blades meeting and, in some instances, greatly exceeding the qualities of razor blades presently used by the public. Although the equipment shown in FIG. 2 may be utilized in production facilities, particularly if modified to contain more than one target and/or more than single blade holding fixtures with commensurate indexing equipment, this type of equipment is not best suited to the high production needs of a large blade manufacturing concern. When considering that in excess of two to three million blades a day must pass through and be subjected to the process outlined in FIG. 1, it can be appreciated that any equipment design intended to en-



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hance the speed of the process and therefore the ultimate output of finished blades is of considerable value and importance. In this regard, it is noteworthy to point out that the addition of certain reactive gases, for example, oxygen, to the sputtering chamber during the refractory sputter deposition steps will under proper conditions greatly increase the sputtering rate and thereby reduce the total sputtering time needed. Any reduction of this nature in the time required for the total process performance when involved in the production of literally millions of blades is of significant import to the overall cost of production of the product. FIG. 3 shows equipment peculiarly suitable to the manufacture of razor blades in accordance with the novel process of the applicants. This equipment permits the continuous sequential batch processing of a large quantity of blades while maintaining extreme limits of cleanliness for the targets and the chambers utilized and further limiting the need for continuous pump-down of the sputter deposition chambers prior to entry of the product into the chamber and subsequent to exit of the product from the chamber.

FIG. 3 shows an exemplary embodiment of an equipment configuration primarily designed for the continuous batch processing of razor blades in conformance with the applicants's process. Chambers 10, 11, 12 and 24' constitute the vacuum chambers necessary for completion of the process. These chambers are joined by vacuum interlocks 21', 22 and 23 between chambers 10, 11 and 12 and 24 respectively. Entrance vacuum and exit vacuum interlocks 21 and 24 are provided for entry of the blades into vacuum chamber 10 and exit of the blades from vacuum chamber 24' respectively. Associated with each chamber is a vacuum system designated as Pump A 27, Pump B 28, Pump C 29, and Pump D 30, which pumps must be capable of producing vacuum levels commensurate with the performance of the process, which levels were previously outlined in the foregoing disclosure and Examples 1 - 3. The blade substrate 20 is shown as moving sequentially through the chambers until its final exit from chamber 24' through the vacuum interlock 24. It is important to note that in a continuous batch system, there is always present in any given chamber a batch of blades mounted on the substrate electrode 20 and only at the beginning and end of any continuous production run are any of the chambers without such blade batch.

Chamber 10 shows within its structure two electrodes 42, 42' surrounded by RF shielding 45, 45' respectively. The two electrodes 42, 42' are provided for the continuous preparation of both edges of double-edge blades or of single-edge blades mounted back-to-back on the substrate holder 20. This contemporaneous treatment of two edges greatly minimizes the time necessary for completion of the process. Vacuum chamber 10 comprises the station in which the sputter etching of the razor blade edges is performed, thereby confining the release of contaminants and the removal of blade edge material to a single chamber thusly preventing any effect of such contamination on the deposition steps of the process. The substrate electrode 20 is shown as connected to the matching impedance unit 33 and is surrounded by an RF shield 49. Vacuum chamber 11 similarly contains two RF electrodes 43, 43' surrounded by their respective shields 46, 46'. The substrate holder 20 is tied to the chamber 11 wall by means of line 51, which chamber 11 is brought to system

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ground as are all the chambers of the system, namely, chambers 10, 11, 12 and 24'. In the instance of chamber 11 both RF electrodes 43, 43' are brought to the matching unit 33 to provide for their RF power excitation. This is contrary to chamber 10 where the two electrodes 42, 42' are brought to the chamber walls by line 50 and 50' respectively, which walls are, as previously indicated, brought to system ground. Targets 40, 40' are shown as fixed to the RF electrodes 43 and 43' respectively in the same manner as the target 57 was attached to the RF electrode 56 in FIG. 2. This chamber 11 is used for the performance of the Deposition I coating and thusly the targets comprise the refractory material or other material to be first applied to the blade edge in order to obtain certain desirable blade characteristics. Proceeding to chamber 12 there is shown a similar equipment arrangement as chamber 11. Mounted in the chamber are RF electrodes 44, 44' with their respective RF shields 47, 47'. Affixed to the face of each electrode are targets 41, 41' comprising the material to be applied in the Deposition II step, i.e., the chromium material or other material displaying the necessary adherence to both the PTFE final lubricious coating and the prior coating applied during the Deposition I step. The substrate electrode 20 is brought to the chamber wall by means of line 52 while RF power is sent to the electrodes by means of connections 37 and 38. In all instances, proper RF connectors and the necessary seals to maintain vacuum are employed to bring lines and connections in and out of the vacuum chambers. Finally we proceed to chamber 24' which is devoid of internal electrode structure as it is only used for an equipment removal purpose. The use of a separate removal vacuum chamber 24' provides for a maintenance of cleanliness in both vacuum chambers 11, 12 as well as a minimization of vacuum pump-down time. The vacuum interlock members 21, 21', 22, 23, 24 essentially comprise sliding valve doors which permit passage of the blade holding members to proceed into and through the sequential chambers until their ultimate exit through the last chamber 24'. It is further important to note at this time that an additional chamber may be inserted between chambers 12 and 24' for sputtering of the lubricious coating if such process step is to be employed, but it is pointed out that the means of application of the lubricious coating is not an essential part of the novel contribution of this invention but rather simply constitutes the process step necessary to the conformation of the ultimate product.

The radio frequency energy again comprises a 13.56 megacycle supply and provides the energy necessary for the electrodes 43, 43', 47, 47' and for substrate electrode 20 in chamber 10. Lines 34-38 are previously indicated supply the RF power to the previously mentioned electrodes and the substrate electrode 20. The switching unit 32 serves to interrupt the RF power supplied by the radio frequency generator 31 when desired and to further either apply RF energy to the various lines or to interrupt it when the particular step of the process so requires. To briefly describe the function of the switching unit, it is pointed out that during the sputter etching step performed in vacuum chamber 10 RF energy is applied to line 34. When the sequential deposition steps are performed in chambers 11 and 12, then the same RF power is applied to the appropriate lines 34-38. The matching impedance unit 33 constitutes separate impedance matching units for each of



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the electrodes involved in the process. No doubt this unit might comprise one single matching unit with various lines or taps brought to the lines 34-38 but, however, it is found most economical and simpler of construction to provide a separate impedance matching unit within the confines of the unit 33 to individually match each of the electrodes during the process step involved.

The DC meter unit 48 is used to monitor the self bias voltage developed during the radio frequency sputtering process on each of the lines 34-38. As heretofore indicated in the specification, each of the electrodes associated with the numbered RF power lines will develop a self bias voltage depending upon the level of power applied and other operating parameters of the system. In addition to the external units now mentioned necessary to the batch process system of FIG. 3, there is also provided for similar purposes as previously outlined with respect to the equipment of FIG. 2 a nitrogen vent unit suitable to purging the chambers and for raising the vacuum level prior to opening of the chambers after evacuation. Argon unit 25 is further provided to supply the ionizable gas to each of the chambers involved in the sputtering process, namely, chambers 10, 11 and 12, and finally a coolant unit 26 passes either water or some other cooling fluid such as ethylene glycol to properly cool the RF electrodes and other members which may be subject to heat problems during the performance of the steps necessary to producing the desired coatings on the razor blade edge.

Briefly to consider the equipment of FIG. 3 in an operational sequence a stack of razor blades held in a fixture similar to that shown in FIG. 6 only deploying blades in opposite directions so that both edges of the blades, in the case of double-edge blades, or complementarily facing blades in the case of single-edge blades 101, are exposed to the sputtering or sputter etching electrodes. The first batch of blades is introduced to the chamber 10 through the vacuum interlock or slide valve 21. Once within this chamber the entire system of chambers 10, 11, 12 and 24' are reduced in vacuum level to approximately  $10^{-6}$  Torr. Argon is then admitted to vacuum chamber 10 by means of the Argon unit 25 which, with the application of RF energy to line 34, results in the formation of a plasma and sputtering of material and contaminants from the exposed edges of the razor blades takes place. Upon completion of this sputter etching operation, vacuum chamber 10 is again pumped down to its  $10^{-6}$  Torr level and the vacuum interlock valve 21' is opened to allow for passage of the razor blades by means of suitable carriers through to vacuum chamber 11. Similar to the just-described sequence of operation for the sputtering etching of the blades in chamber 10, Argon by means of Argon unit 25 is admitted to the vacuum chamber 11 with the application of RF energy to the lines 35, 36. Again, a plasma results. However, in this instance, since the electrodes 43, 43' and their targets 40 and 40' respectively are now brought to the RF power, the sputtering takes place from the target onto the blade edges. Since the electrodes to which RF energy is applied take on the negative self biasing voltage, the positive ions created in the plasma by collisions with the energetic electrons are attracted toward the targets 40, 40', thereby causing the removal of material from their surfaces and their resultant energetic deposition upon the intersecting surfaces forming the blade edges. Once

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again the chamber 11 is evacuated to the  $10^{-6}$  Torr range and the associated vacuum interlock valve 22 is opened for passage of the blades through to the chamber 12.

The same steps are performed in chamber 11 for the Deposition I process are repeated in chamber 12 for the Deposition II process, thereby resulting in a blade having two coatings placed over its ultimate edge and the facets or surfaces forming such ultimate edge. Subsequent to this last sputter deposition step, chamber 12 is evacuated to the  $10^{-6}$  Torr level and the blades are passed through vacuum interlock valve 23 to the last chamber 24'. It should be pointed out at this time that each of the valves 21, 21', 22, 23 close after passage of the blades through to the next chamber. With the blades in vacuum chamber 24' this chamber is vented by means of the nitrogen vent unit 39 to atmospheric level and vacuum interlock valve 24 is opened for removal of the blades from the system. As previously pointed out, as blades are removed from one chamber to the next, new blades are being introduced from the chamber going before, thus constituting a continuous batch sequential processing system. The operating parameters, i.e., vacuum, time, power, self bias voltage, are substantially the same as those indicated in Examples 1-3 and the description going before such examples, the difference being that each step in the operation is performed in a separate chamber rather than a single chamber requiring frequent opening and closing of the system with resultant susceptibility to contamination. Operation of equipment such as this is well known to those individuals ordinarily skilled in the art once the essential operating parameters and conditions are brought to their attention. It is pointed out that U.S. Pat. application Ser. No. 861,937, filed Sept. 29, 1969, adequately describes and discloses a system appropriate to the carrying out of the continuous batch process herein disclosed. It would be only necessary to alter the target materials and operating parameters to conform to those novel aspects of the applicant's invention.

In considering both the continuous batch process of FIG. 3 and the single batch operating equipment and procedure demonstrated in FIG. 2, it is important to indicate its applicability to band razor blades, which comprise continuous strips of predetermined length commensurate with a certain number of shaving edges normally provided on discreetly dimensioned razor blades. In use, such continuous strips are indexed a certain length substantially equaling a single-edge length of a discreet dimensioned razor blade. When depositing coatings, or, more precisely, sputter depositing coatings, on such edges a long continuous length of band razor blade is utilized, often comprising lengths constituting miles or substantial portions of miles in length. A copending application Ser. No. 144,510, filed May 18, 1971, describes a single target electrode configuration capable of sputter depositing coatings on a band razor steel blade. However, due to the limited dimensions of the target, the band blade must be rotated under the target during the sputtering process, thusly seriously hampering the efficiency and output of the process. While the equipment of FIG. 2 would still require rotation of the band razor under the target 57 in order to obtain a reasonably uniform and continuous sputter deposit coating due principally to the limitations on dimensions of such chambers, the continuous batch system of FIG. 3 is capable of much more effi-



cient operation with respect to this type of razor blade.

FIGS. 7, 8 and 9 show an exemplary fixture capable of holding continuous strips of band blade during the sputtering process. Due to the large target configurations which may be employed in the chambers 10, 11, 12 of the continuous batch system, it is possible to place a complete spiral of band razor steel in facing relationship to the sputtering targets, thereby obviating the need for rotation of the blade edge transversely across the face of the target during the sputtering process or operation. It has been found to be most advantageous to place in the fixture shown in FIGS. 7, 8 and 9 more than a single spiral of band razors and to place them in oppositely facing directions so as to take advantage of the dual electrode configuration of FIG. 3. Thusly, it is possible to greatly increase the efficiency and improve the uniformity of the sputtering process on band razor blades in that a total of four stationary spirals may be coated at one time as opposed to a single spiral being rotated beneath a target as previously or heretofore utilized. The fixture for holding the band razor strip constituting two nests into which the strip is placed in spiral configuration, which nests are then clamped together in oppositely facing directions by means of a ring clamp. It has been found that the nesting of the blades exposing no more than 0.005 of an inch over the upper surface of the nest holder 110 is essential to providing a uniform coating on the surfaces forming the blade edge. It has also been determined that while of less criticality than the exposure of the edge of the upper surface 110, the dimensions of the inner hub indicated by diameter D1 and the outer hub indicated by diameter D2 are of significance. Generally it is preferred that the diametrical distance from the outside of the periphery formed by diameter D2 to the outermost edge of the blade spiral should most appropriately be between one and two inches and that the inner hub be approximately between 5 and 9 inches in diameter. Of course, these dimensions may be altered depending upon the amount of blade steel desired to be contained in the blade spiral and the dimensions of the sputtering chambers. The nests 110 in combination with the band clamp 111 are in turn captured within a second fixture 112 for actual placement within the continuous batch system. The holder 112 is then carried by appropriate motion drives through the various chambers for application of the desired coatings.

Referring to Example 1 heretofore set forth, it has been found that the material sputtered upon the substrate surface in the Deposition I step comprises a crystalline structure of hexagonal lattice form having a preferred orientation. It has also been found through the application of microprobe analysis, i.e., the examination of emitted X-rays upon subsection of the material to an electron beam, that the material constitutes in its elemental forms pure  $Al_2O_3$  within the precision limits of the microprobe equipment. Relating these two factors as to the morphology of the coating and the purity of the constituents, the material sputtered from the target 57 onto the substrate apparently constitutes the same synthetic sapphire of which the target is composed. Thus, in addition to disclosing a novel method for the application of refractory materials to razor blades or, more generally, cutting edges and the subsequent preparation of such surfaces for the lubricious material, there is further presented in accordance with the invention a novel process for applying a coating of

corundum or synthetic sapphire to the surface of a substrate. While not fully understanding the mechanism of this material transfer by means of sputter deposition, it is presumed that the energies of the atomic size particles or molecules removed from the surface of the target 57 are within the range necessary to bring about the desired crystalline formation on the surface of the substrate. Thus, there is completely transferred to the substrate the characteristics of the refractory target material commensurate with a thin film formed of such material. Thus, this wholly unexpected result of the described blade manufacturing process finds ready application for other purposes. It would now seem possible to transfer sapphire or other material through sputter deposition means from a target to a substrate, which substrate may comprise any equipment on which such refractory coatings would be suitable either for wear, dielectric or other suitable and appropriate reasons.

It is apparent that the blade material may be composed of material such as carbon steel, chromium steel, tungsten steel, molybdenum steel or chrome-nickel steel. Further, it is obvious that the blade material may be an alloy containing material selected from the group consisting of stainless steel, carbon steel, chromium steel, tungsten steel, molybdenum steel, and chrome-nickel steel.

In summary, the disclosure of this application has set forth a novel process for the production of razor blades, or more generally speaking cutting edges, having wholly unanticipated and unpredictable qualities. It is emphasized that the teachings of this disclosure are intended to be illustrative and exemplary of the invention and not to be delimiting of its scope. Thus, it is intended that those variations and modifications of the novel process and products produced thereby which would become obvious to one ordinarily skilled in the art are to be considered within the scope and ambit of the applicants' invention.

What is claimed is:

1. A method of making a razor blade comprising the steps of:

forming a blade from a suitable material, the blade having an elongate edge comprising two intersecting surfaces;

sputter depositing on the edge a first coating of refractory material;

coating the edge with a second material displaying adhesion to a subsequent coating of lubricious material and to the refractory material; and then coating the edge with the lubricious material.

2. The method of claim 1 wherein the first coating is RF sputter deposited and the second material is sputter deposited.

3. The method of claim 2 wherein the refractory material is corundum.

4. The method of claim 2 wherein the refractory material is selected from the group consisting of glass, corundum, quartz, alumina, beryllia, silicon carbide, boron nitride, and tungsten carbide.

5. The method of claim 4 wherein the refractory material comprises alloys and mixtures of materials selected from the group.

6. The method of claim 2 wherein the refractory material is synthetic sapphire.

7. The method of claim 1 wherein the total thickness of the deposited refractory material and the second ma-



terial is limited to that necessary to maintain a desired degree of edge sharpness.

8. The method of claim 7 wherein the total thickness is approximately 500 Angstrom units.

9. The method of claim 7 wherein the thickness of the refractory material is approximately 300 Angstrom units and the thickness of the second material is approximately 25 Angstrom units.

10. The method of claim 2 wherein the RF sputter depositing comprises the steps of:

disposing the blade in an evacuated chamber having an electrode on which is mounted a target of refractory material;

introducing into the chamber an ionizable gas and establishing a plasma by imposing an RF potential between the electrode and the blade;

depositing on the edge particles dislodged from the target by impingement of gas ions formed in the plasma upon collision of RF excited electrons and the ionizable gas molecules.

11. The method of claim 10 wherein the chamber is evacuated to approximately  $10^{-6}$  Torr and the ionizable gas is introduced to a pressure of approximately between 5 and 8 (10)<sup>-5</sup> Torr.

12. The method of claim 11 wherein the refractory material is synthetic sapphire, the ionizable gas is Argon and the blade is positioned approximately 2 inches from the target, the edge apex being disposed substantially in a plane parallel to the target and the refractory material is deposited at a rate of approximately 30 Angstroms per minute for a period between approximately 5 and 10 minutes.

13. The method of claim 12 wherein the frequency is 13.56 MC and wherein the blade is sputter etched prior to deposition of the refractory material and a shutter is interposed between the target and the blade during the step of sputter etching.

14. The method of claim 13 wherein capacitor means is serially connected between the blade and the RF potential and the shutter is connected to ground during sputter etching and wherein when the shutter is removed, the RF potential is connected to the electrode and the blade is connected to ground for sputter deposition.

15. The method of claim 14 wherein the target is pre-cleaned prior to sputter etching and wherein during pre-cleaning the shutter is interposed, the RF potential connected to the electrode and the shutter is connected to ground.

16. The method of claim 15 wherein the following parameters are maintained during pre-cleaning, sputter etching and sputter deposition, respectively:

Power	approximately 400 W, 1.4 KW and 1.4 KW;
Self bias electrode voltage	approximately 1 KVDC, 2.2 KVDC and 2.2 KVDC and
Time	approximately 5 min., 1 min. and between 5 and 10 min.

17. The method of claim 10 wherein the second material is RF sputtered in accordance with the steps of claim 10.

18. The method of claim 17 wherein the second material is deposited to a thickness sufficient to provide adhesion of the subsequent lubricious material.

19. The method of claim 18 wherein the second material is deposited to a thickness of approximately 25 Angstrom units and the second material is a metal containing material.

20. The method of claim 19 wherein the second material is selected from the group consisting of chromium, platinum, aluminum, titanium and iron.

21. The method of claim 19 wherein the second material comprises mixtures and alloys of metals selected from the group consisting of chromium, platinum, aluminum, titanium and iron.

22. The method of claim 19 wherein the second material is chromium.

23. The method of claim 19 wherein the lubricious material is a polymer material.

24. The method of claim 23 wherein the polymer is selected from the group consisting of polytetrafluoroethylene, polypropylene, polyhexafluoropropylene, polychlorotrifluoroethylene and polyethylene.

25. The method of claim 23 wherein the lubricious material comprises copolymers and telomers of polymers selected from the group consisting of polytetrafluoroethylene, polypropylene, polyhexafluoropropylene, polychlorotrifluoroethylene and polyethylene.

26. The method of claim 23 wherein the polymer is polytetrafluoroethylene.

27. The method of claim 6 wherein the lubricious material is sputter deposited onto the edge.

28. The method of claim 27 wherein the lubricious material is deposited to a thickness of at least 1,000 Angstrom units.

29. The method of claim 17 wherein the edge is sputter etched in a first vacuum chamber, the blade is moved through a vacuum interlock to a second vacuum chamber in which the edge is sputter deposited with the refractory material, the blade is then moved through a second vacuum interlock to a third vacuum chamber in which the edge is sputter deposited with the second material, and finally the blade is moved through a third vacuum interlock to a fourth vacuum chamber which is then vented to the atmosphere to permit blade removal for subsequent coating with the lubricious material.

30. The method of claim 29 wherein blades are continuously sequentially passes through the chambers.

31. The method of claim 1 wherein the refractory material comprises an aluminum oxide compound formed on the elongate edge when material sputtered from an aluminum target combines with oxygen present in the environment.

32. The method of claim 10 wherein the sputtering rate is increased by the presence of a reactive gas.

33. A cutting instrument comprising:  
an elongate edge of narrow included angle formed by two intersecting surfaces of a refractory material, an overlay coating of material over the edge for providing adhesion to the refractory material and a lubricious material, and  
a final coating of the lubricious material.

34. The cutting instrument of claim 33 wherein the refractory material and the coating are sputter deposited on the edge.

35. The cutting instrument of claim 33 wherein the refractory material is synthetic sapphire.

36. The cutting instrument of claim 34 wherein the refractory material is selected from the group consist-



ing of corundum, alumina, glass, quartz, beryllia, silicon carbide, tungsten carbide and boron nitride.

37. The cutting instrument of claim 35 wherein the refractory material is RF sputter deposited on the edge.

38. The cutting instrument of claim 37 wherein the overlay coating is RF sputter deposited.

39. The cutting instrument of claim 38 wherein the lubricious material is RF sputter deposited.

40. The cutting instrument of claim 37 wherein the total thickness of the refractory material and the overlay coating does not exceed approximately 500 Angstrom units.

41. The cutting instrument of claim 40 wherein the thickness of the refractory material is approximately 300 Angstrom units and the thickness of the overlay coating is approximately 25 Angstrom units.

42. The cutting instrument of claim 41 wherein the intersecting surfaces are honed surfaces and the narrow included angle is less than approximately 30°.

43. The cutting instrument of claim 42 wherein the narrow included angle is approximately 20°.

44. The cutting instrument of claim 43 wherein the cutting instrument material is stainless steel.

45. The cutting instrument of claim 43 wherein the cutting instrument material is selected from the group consisting of stainless steel, carbon steel, chromium steel, tungsten steel, molybdenum steel, and chrome-nickel steel.

46. The cutting instrument of claim 43 wherein the cutting instrument material is an alloy containing material selected from the group consisting of stainless steel, carbon steel, chromium steel, tungsten steel, molybdenum steel, and chrome-nickel steel.

47. A method for applying a lubricious material to a cutting instrument having an edge formed by two intersecting surfaces of a refractory material having limited adhesion to the lubricious material comprising the steps of:

sputter depositing on the edge an overlay coating displaying adhesion to the lubricious material; and then coating the edge with the lubricious material.

48. The method of claim 47 wherein both the refractory material and the overlay coating material are sputtered on the edge and the refractory material is synthetic sapphire.

49. The method of claim 48 wherein both the refractory material and the overlay material are RF sputtered on the edge.

50. The method of claim 49 wherein the refractory material is selected from the group consisting of glass, quartz, corundum, alumina, beryllia, silicon carbide, tungsten carbide and boron nitride.

51. The method of claim 49 wherein the refractory material is an aluminum oxide compound formed by sputter depositing aluminum in an oxygen atmosphere.

52. The method of claim 49 wherein the lubricious material is selected from the group consisting of polytetrafluoroethylene, polypropylene, polyhexafluoropropylene, polychlorotrifluoroethylene and polyethylene.

53. The method of claim 52 wherein the lubricious material is sputter deposited on the overlay coating.

54. The method of claim 49 wherein the overlay coating is a metal containing material.

55. The method of claim 54 wherein the metal is selected from the group consisting of chromium, platinum, titanium, aluminum and iron.

56. The method of claim 54 wherein the overlay coating is an alloy containing metal selected from the group consisting of chromium, platinum, titanium, aluminum and iron.

57. The method of claim 54 wherein the overlay coating material is chromium.

58. The method of claim 54 wherein the overlay coating thickness is approximately 25 Angstrom units and the total thickness of both the refractory material and the overlay coating is limited to that necessary to maintain a desired degree of edge sharpness.

59. The method of claim 58 wherein the refractory material coating is approximately 300 Angstrom units in thickness.

60. The method of claim 32 wherein said reactive gas is oxygen.

61. The cutting instrument of claim 33 wherein the cutting instrument is a razor blade.

62. The cutting instrument of claim 35 wherein the cutting instrument is a razor blade.

63. The cutting instrument of claim 36 wherein the cutting instrument is a razor blade.

64. The cutting instrument of claim 37 wherein the cutting instrument is a razor blade.

65. The cutting instrument of claim 38 wherein the cutting instrument is a razor blade.

66. The cutting instrument of claim 40 wherein the cutting instrument is a razor blade.

\* \* \* \* \*

# EXHIBIT E



# **HANDBOOK OF PHYSICAL VAPOR DEPOSITION (PVD) PROCESSING**

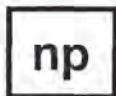
**Film Formation, Adhesion, Surface  
Preparation and Contamination Control**

by

**Donald M. Mattox**

Society of Vacuum Coaters  
Albuquerque, New Mexico

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### ***Atomistic Film Growth and Growth-Related Film Properties 487***

Single crystal overgrowth can be accomplished with large mismatches in lattice parameters between the film and substrate either by keeping the thickness of the deposited material small so that the mismatch can be taken up by straining the film lattice without forming lattice defects (“strained layer superlattice”), or by using a “buffer” layer to grade the strains from the substrate to the film. For example, thick single crystal SiC layers can be grown on silicon by CVD techniques even though the lattice mismatch is large (20%).<sup>[236]</sup> This is accomplished by forming a buffer layer by first carbonizing the silicon surface and then grading the composition from the substrate to the film. However, in general, if the lattice mismatch is large, the interface has a high density of dislocations and the resulting film will be polycrystalline.

Energetic adatoms and low energy ion bombardment during deposition can be used as a partial substitute for increased substrate temperature in epitaxial growth process. Carefully controlled bombardment can lower the temperature at which epitaxy can be obtained.<sup>[10][237]</sup> This is probably due to increased surface mobility of the adatoms. Ion beams of the depositing material (“film ions”) have also been used to deposit epitaxial films.<sup>[238]</sup>

Oriented growth can be enhanced by “seeding” of the substrate surface with oriented nuclei. Such “seeds” can be formed by depositing a small amount of material, heating the surface to form isolated oriented grains and then using these grains as seeds for the deposition of an oriented film at a lower temperature.<sup>[239]</sup>

### **Amorphous Film Growth**

Amorphous materials are those that have no detectable crystal structure. Amorphous film materials can be formed by:

- Deposition of a natural “glassy” material such as a glass composition<sup>[240][241]</sup>
- Deposition at low temperatures where the adatoms do not have enough mobility to form a crystalline structure (quenching)<sup>[101]</sup>
- Ion bombardment of high modulus materials during deposition<sup>[242]</sup>
- Deposition of materials some of whose bonds are partially saturated by hydrogen—examples include a-Si:H, a-C:H, and a-B:H.<sup>[81][82]</sup>

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- Sputter deposition of complex metal alloys<sup>[243]</sup>
- Ion bombardment of films after deposition<sup>[244]</sup>

### **Metastable or Labile Materials**

Metastable or labile phases are phases of materials that are easily changed if energy is available for mass transport processes to occur. Deposition processes can allow the development of metastable forms of the material. Metastable crystal structures can be formed by rapid quenching of high temperature phases of the deposited material or can be stabilized by residual stresses or impurities in the film. For example, diamond which is a metastable phase of carbon, is formed naturally in a high pressure and temperature environment, and changes to graphitic carbon on heating. However, diamond films can be deposited using the proper low-temperature vacuum deposition techniques (Sec. 9.7.8). Metastable film compositions can be formed under deposition conditions that do not allow precipitation of material when it is above the solubility limit of the system. For example, concurrent low energy ion bombardment using "dopant ions" allow doping of semiconductor films to a level greater than can be obtained by diffusion doping techniques.<sup>[245]</sup>

### **9.4.8 Gas Incorporation**

Bombardment of a surface with gaseous ions during film growth or sputter cleaning can incorporate several atomic percent of gas in the near-surface region. Bombardment of the growing film by a gaseous species can result in the gas being incorporated into the bulk film since the surface is being continually buried under new film material. This effect is similar to the process of inert gas pumping in a sputter-ion pump. Very high concentrations of normally insoluble gases can be incorporated into the film structure.<sup>[246][247]</sup> For example, up to 40 at% hydrogen and helium can be incorporated into gold films. Using  $\text{He}^3$  and NMR techniques it was shown that the helium is atomically dispersed but can be caused to agglomerate into voids on heating.<sup>[248]</sup>

To prevent gas incorporation in the surface or growing film, the surface can be heated to desorb the gases before they are covered over or the bombardment energy can be less than a few hundred eV which will



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Composite materials of metal particles in a polymer matrix can be formed by deposition of the metallic phase during plasma polymerization. Such a composite film has been shown to have a better wear durability than the polymer film alone<sup>[352]</sup> and to have interesting optical properties.<sup>[353]</sup>

### **9.7.7 Intermetallic Films**

Intermetallic compounds are formed from electropositive and electronegative metals which chemically bond to form compounds with a specific composition and crystalline structure. Intermetallic films are often formed by depositing the film material on a hot surface so that the adatoms diffuse and react with the surface material converting it into a silicide, aluminide, etc. Very corrosion resistant intermetallic films can be formed by co-deposition processes at high temperatures. These include the very chemically-stable compounds  $\text{Mo}_5\text{Ru}_3$  and  $\text{W}_3\text{Ru}_2$ <sup>[354]</sup> and  $\text{ZrPt}_3$  and  $\text{ZrIr}_3$  which are d-orbital bonded intermetallic compounds.<sup>[355]–[357]</sup>

### **9.7.8 Diamond and Diamond-Like Carbon (DLC) Films**

Recently great progress has been made in the deposition of diamond and diamond-like carbon (DLC) coatings for industrial applications.<sup>[358]</sup> Natural diamond with its high hardness, low coefficient of friction, high thermal conductivity, good visible and infrared transparency, and chemical inertness has long provided a goal for the thin film deposition community.

Diamond is a carbon material with a specific crystallographic structure (diamond structure) and specific chemical bonding ( $\text{sp}^3$  bonding). Diamond-like carbon (DLC) is an amorphous carbon material with mostly  $\text{sp}^3$  bonding that exhibits many of the desirable properties of the diamond material. The DLC material is sometimes called “amorphous diamond”—an oxymoron that should be avoided.

The property of the carbon  $\text{sp}^3$  bonding that allows the deposition of both diamond and DLC coatings, is its relative chemical inertness to hydrogen reduction. If the  $\text{sp}^3$  bond is formed during deposition, then the carbon film is stable to hydrogen etching. If, however, the  $\text{sp}^2$  (graphite) bond is formed, the material is much more susceptible to hydrogen etching.

Polycrystalline diamond films are formed if the deposition temperature is high enough ( $>600^\circ\text{C}$ ) to allow atomic rearrangement during

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deposition. DLC films are formed at lower temperatures (room temperature and even below) where the atoms cannot arrange themselves into the diamond structure giving an amorphous material. The DLC films can have varying amounts of  $sp^2$  bonding and included hydrogen, which affect their properties. The  $sp^3$ -bonded material can be deposited by a number of techniques most of which involve "activating" both a hydrocarbon species, such as methane, to allow carbon deposition, and hydrogen to provide the etchant species.

Polycrystalline diamond films are most often deposited by a hot filament technique using a chemical vapor precursor (HFCVD), a combustion flame technique, or a Plasma Enhanced CVD (PECVD) technique using an rf (13.56 MHz) or microwave (2.45 GHz) plasma. In the hot filament process, the hot surface dissociates the gases, while in the flame process, the gases are dissociated in a reducing (hydrogen-rich) flame. In the plasma process, the gases are dissociated and ionized in the plasma. In all cases, the diamond film that is formed is polycrystalline and has a rough surface. This is due to the method of film nucleation on the substrate surface and the nature of the film growth. The rough surface has a high coefficient of friction and a great deal of development work is being done to try to improve the surface smoothness for wear and friction applications. The physical and chemical properties of the deposited polycrystalline films approach those of natural diamond. Free-standing diamond structures can be fabricated by etch-removal of the substrate after deposition. Using a microwave technique, researchers have produced diamond films having a thermal conductivity of 8 watts/cm deposited at rates of 10 micron/hour for a cost of about \$50/carat (200 mg).

DLC films are made primarily using PECVD and single or dual ion beam techniques at low substrate temperatures. DLC films are smooth with most properties approaching those of natural diamond, with the exception of thermal conductivity which is much lower for DLC films than for natural diamond. The dual beam technique, which uses separate hydrogen-derived and methane-derived ion beams of about 125 eV ion energies, produces films that have the highest index of refraction and the lowest optical absorptance of all the low-temperature DLC deposition techniques. Thin (1500 Angstroms) DLC films are being used as abrasion-resistant coatings on infrared optics and optical products such as eyeglasses, sunglasses, and scanner windows. NASA researchers report that 1000 Å dual beam-deposited DLC films transmit 85% of light at 0.5 microns wavelength.



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When techniques for producing smooth, adherant diamond films are developed, it is expected that they will have extensive application in the semiconductor packaging industry because of diamond's high thermal conductivity (about 5 times that of copper) and high electrical resistivity. Diamond can also be used as a cold cathode electron emitter and as such is of interest in the flat-panel display industry. Diamond films may also provide protection to surfaces in low-earth orbit where oxygen erosion is a problem.

#### **9.7.9 Hard Coatings**

Hard coatings, formed by reactive Physical Vapor Deposition (PVD) processes, are becoming widely used in the decorative coating and tool industries.<sup>[346]</sup> Hard decorative PVD coatings are more resistant to wear and corrosion than are electroplated decorative coatings, such as gold and brass, which must use a polymer topcoat for protection. Such decorative hard coatings are being used on plumbing fixtures, sporting goods, metal dinnerware, eyeglass frames, door hardware, and other such applications where the coating is subjected to wear, abrasion, and corrosion during use and cleaning. Titanium nitride (TiN) is used for a gold-colored coating and zirconium nitride (ZrN) looks like brass. Titanium carbonitride ( $\text{TiC}_x\text{N}_y$ ) can have a color which varies from bronze to rose to violet to black depending on the composition. The titanium carbonitride coatings are generally harder than the nitride coatings. Aluminum can be added to the nitrides to impart some high temperature oxidation-resistance. Chromium carbide (CrC) coatings have a silver color and are hard and oxidation resistant.

In order to get the most hard, dense, wear and corrosion resistant coating, the substrate temperature should be as high as possible and concurrent bombardment by energetic atomic-sized particles during the reactive deposition should be used (Fig. 6-11). When coating temperature-sensitive substrates such as plastics, the temperature must be kept low and concurrent bombardment can be used to densify the film. One technique for coating temperature-sensitive materials, uses the deposition of many thin layers separated by a cooling period. This is done by mounting the parts on a rotating fixture that is passed in front of the deposition source, multiple times. In one decorative application, multiple, alternating gold and TiN layers are deposited, using the same type of fixture. In this application, as the gold wears off at high points it exposes the underlying gold-colored TiN and the coating still looks gold and the article can be advertised as being gold plated.

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## Glossary of Terms and Acronyms used in Surface Engineering

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*Abnormal glow discharge* (plasma)—The DC glow discharge where the cathode spot covers the whole cathode and an increase in the voltage increases the cathode current density. This is the type of glow discharge used in most plasma processing. See *Normal glow discharge*.

*Abrasion test* (characterization)—Testing a film adhesion and abrasion resistance by rubbing, impacting or sliding in contact with another surface or surfaces. Examples: tumble test, tabor test, eraser test.

*Abrasive* (cleaning)—A material, such as a particle or a rough solid, that is capable of removing material from a surface when there is pressure and movement between the material and the surface.

*Abrasive cleaning*—The removal of surface material (gross cleaning), including contamination, by an abrasive action.

*Abrasive compound*—A material used to remove material from a surface by abrasion. Surface smoothness after abrasion is a secondary consideration. Examples: silicon carbide, emery, silica, alumina. See *Polishing compound*.

*Abrasive flow machining* (vacuum technology)—A means of smoothing a surface using a slurry of abrasive particles in a fluid that is passed over the surface. Also called *slurry polishing*.

*Abrasive transfer, contamination by* (cleaning)—Transfer of material to a clean surface by contact or friction with a material to which it adheres such as a polymer on a high surface energy surface or chromium on a clean oxide surface.

*Abrupt-type interface* (film formation)—The interface that is formed between two materials (A and B) when there is no diffusion or chemical compound formation in the interfacial region. The transition of A to B in the length of a lattice parameter ( $\approx 3\text{\AA}$ ). See *Interface*.



## Glossary 767

*Aliphatic solvent* (cleaning)—A type of solvent that consists of straight-chain hydrocarbons such as hexane and naphtha.

*Alkaline cleaner* (cleaning)—A basic cleaner that cleans by saponifying of oils and chelating inorganic soils. The cleaner can also have agents for surfactants for emulsifying, wetting and penetrating, alkaline builders for neutralizing water hardness interference, corrosion inhibitors, etc. Alkaline cleaning is often followed by an acid rinse to neutralize the adhering alkaline material and remove non-soluble precipitates formed by reaction with the alkaline material. A mild alkaline cleaner has a pH of about 9.5 to 10.0, a strong alkaline cleaner will have a pH of 13.0.

*Alloy*—A solid solution where there is a stable mixture of the materials.

*Altered region* (ion bombardment)—The region near the surface which has been altered by the physical penetration of the bombarding species or by “knock-on” lattice atoms. In the extreme case this can lead to the amorphization of the region. See *Near-surface region*.

*Alternating ion plating* (film deposition)—A repetitious process where a few monolayers of condensable film material is deposited and then the surface is bombarded followed periodically by more deposition and more bombardment. Also called *pulsed ion plating*.

*Alumina* (substrate)—Aluminum oxide ( $\text{Al}_2\text{O}_3$ ). Alumina substrates are usually in the form of sintered material with some amount (4-15%) of silica glassy phase.

*Aluminize*—The process of depositing aluminum on a surface from a liquid or vapor.

*Aluminize*—The process of reacting a surface with aluminum to form an aluminum alloy or intermetallic phase.

*Ambient conditions* (vacuum technology, contamination control)—Conditions such as pressure, air composition, temperature, etc., that are present in the processing area.

*Amine*—Any one of a group of organic compounds derived from ammonia ( $\text{NH}_3$ ) by replacement of one or more hydrogen atoms by organic radicals.

*Ammonia* ( $\text{NH}_3$ )—A chemical precursor vapor for nitrogen that is easier to decompose than is  $\text{N}_2$ .

*Amorphous* (crystallography)—A material without a periodic structure that would be revealed by x-ray diffraction. This is typically a grain size of less than about 30 Å.

*Ampere* (*A*)—Electrical current of one coulomb ( $1.6 \times 10^{19}$  electrons) per second. Also called an *Amp*.

*Amphoteric material*—A material that can either gain or lose an electron (i.e., act as either an acid or a base) in a chemical reaction. Example: aluminum can form  $\text{Al}_2\text{Cu}$  or  $\text{Al}_2\text{O}_3$ .

*Amorphous* (crystallography)—Material with a grain size so small (<30 Å) that the x-ray diffraction pattern does not show any crystallinity. See *Glassy*.

*Angle-of-incidence* (film formation)—The angle of impingement of the depositing adatom flux as measured from the normal to the surface.

*Angle-of-incidence effect* (film growth)—The effect of angle-of-incidence of the adatoms on the development of a columnar morphology.

*Angstrom* (*Å*)—A unit of length equal to  $10^{-10}$  meters or 0.1 nanometer.

*Anhydrous* (cleaning)—Without water. Example: anhydrous (absolute) alcohol.

*Anion* (electroplating)—An ion that is negatively charged and will move toward the anode.

## Glossary 807

*Gas incorporation* (film formation)—Incorporation of soluble or insoluble gases during film growth either by physical trapping or by low-energy implantation by bombarding species. Example: incorporation of helium in gold films. See *Charging, hydrogen*.

*Gas scattering*—Scattering of a high velocity atom by collision with gas molecules. See *Thermalization, Gas scatter plating*.

*Gas scatter plating* (film deposition)—Increasing the throwing power of the depositing atoms by scattering the atoms in a gaseous atmosphere. Does not work very well without a plasma due to gas phase nucleation and the deposition of ultrafine particles. When a plasma is present the ultrafine particles become negatively charged and do not deposit on the substrate particularly if the substrate is at a negative potential as in ion plating.

*Gas-phase nucleation* (particle formation)—The nucleation of atoms in a gaseous environment where multi-body collisions allow the removal of the energy released on condensation. See *Gas evaporation*.

*Gaseous arc*—An arc formed in a chamber containing enough gaseous species to aid in establishing and maintaining the arc. See *Vacuum arc*.

*Gasket* (vacuum technology)—The object between sealing flanges that deforms or shears, thus creating the vacuum-tight seal. See *Flange*.

*Gate valve* (vacuum technology)—A mechanical sealing valve where the motion of the sealing plate is mostly parallel to the plane of the seal. Generally the valve opening is round so that the maximum opening is achieved with the use of the least sealing area. See *Vacuum valves*.

*Gauge*—A measuring device. Example: vacuum gauge. See *Sensors*.

*Gauge*—A thickness unit. Example: 18 gauge steel sheet.

*Gauge*—A diameter unit. Example: 12 gauge electrical wire.

*Gauge band* (web coating)—A continuous lane of film in the machine direction of the roll that is abnormally thick (hard band) or thin (soft band).

*Gauss*—Unit of magnetic field intensity equal to one Maxwell/cm<sup>2</sup> or 10<sup>-4</sup> Weber/m<sup>2</sup>. See *Oersted* (cgs system), *Tesla* (SI system).

*Getter* (vacuum technology)—A material that will react with or adsorb reactive gases in the vacuum environment.

*Getter* (vacuum technology)—To remove gases either by a chemical reaction so as to form non-volatile solid species containing the gas, or by absorption of the gases in the getter material.

*Getter pump* (vacuum technology)—A vacuum pump that operates by reaction of a surface with the gaseous species to form a non-volatile reaction product or by absorption of the gases into the bulk of a getter material. In reaction-type getter pumps the getter materials are often deposited by evaporation or sublimation. Adsorption-type getter pumps are sometimes called non-evaporative getter pumps. See *Vacuum pump*.

*Getter pumping, during deposition* (PVD technology)—The gettering action that accompanies the deposition of a reactive film material such as titanium in an oxygen environment.

*Gilding*—Overlaying a surface with a very thin free-standing film (e.g. gold or silver) which is adhesively bonded to the surface or held to the surface by electrostatic forces.

*Glass* (substrate)—A non-crystalline material. Generally composed of a mixture of oxides and additives (glass formers) that inhibit crystallization.

*Glass, float* (substrate)—Glass sheet formed by continuously pouring molten glass on a bed of molten tin. Most window glass is made by this technique which leaves a layer of tin oxide on one surface.